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PRELIMINARY

Screening Survey for Metals and Dioxins in Fertilizers, Soil Amendments, and Soils in Washington State

WASHINGTON STATE DEPARTMENT OF ECOLOGY

WSDA WASHINGTON STATE DEPARTMENT OF AGRICULTURE

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Screening Survey for Metals and Dioxins in Fertilizers, Soil Amendments, and Soils in Washington State

by

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* These *Supplemental Appendices* are included in a companion document, Ecology Publication No 98-332 For ordering information, see the back of the front cover of this report

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Glossary

For purposes of this report, the following definitions are used*

Bioaccumulative chemicals of concern – a category of contaminants that are toxic, long-lived, and can accumulate in organisms **

Bulk fertilizer – fertilizer available in unpackaged form for agricultural application

By-product – a substance that is not one of the primary products of a production process.

Contaminant – any substance that does not occur naturally or occurs at concentrations greater than natural background levels.

Dioxins – in this report, refers to polychlorinated dibenzo-*p*-dioxins and polychlorinated dibenzofurans with chlorine atoms in the 2,3,7,8, positions of the molecule

Fertilizer – any substance containing one or more recognized plant nutrients and which is used for its plant nutrient content and/or which is designated for use or claimed to have value in promoting plant growth, and includes commercially valuable concentrations of nitrogen, phosphoric acid, available phosphorus, potash, calcium, magnesium, or sulfur, limes, gypsum, manipulated animal and vegetable manures.

Fertilizer products – in this report, refers to all fertilizers and related products sampled These products include bulk agricultural fertilizers, home-use fertilizers, micronutrients, and soil amendments

Hazardous substance – any dangerous or extremely hazardous substance as defined in RCW 70 105 and other applicable regulations In general, substances determined to present a threat to human health and the environment

Micronutrient – a trace plant nutrient or minor element (other than a primary nutrient) such as boron, chlorine, cobalt, copper, iron, manganese, molybdenum, sodium, or zinc

Natural background – the concentration of a substance consistently present in the environment which has not been influenced by localized human activities

Nutrient – an element required for normal growth and development of plants or animals

Relative percent difference – a measure of precision, it is the ratio of the difference and the mean of the results expressed as a percentage A low RPD indicates high precision

Soil amendment – any of various organic or inorganic materials added to soil to affect its physical properties

Solid waste – all liquid, solid, and semi-solid materials which are not the primary products of public, private, industrial, commercial, mining and agricultural operations

Tag-along – an unintended or unnecessary element or substance found in a product

Toxic – an element or substance that is or has potential to be harmful to human health or the environment.

Toxic equivalent – the sum of all TEFs

Toxic equivalent factors – the relative toxicity value of different types of dioxins.

Waste-derived – a waste or by-product from any industnal process that is recycled into fertilizer or soil amendments

* Many of these definitions are codified in the Washington Administrative Code

** Socha, A C et al , 1993 Candidate Substances for Bans, Phase-outs, or Reductions – Multimedia Revision Ontario Ministry of Envıronment and Energy ISBN)-7778-0774-2

Acronyms and Abbreviations

BCC –	bioaccumulative chemicals of concern
CEC –	cation exchange capacity
CKD –	cement kiln dust
d –	day
DL –	detection limit
DTPA –	diethylenetriaminepentaacetic acid extractions
dw –	dry weight
Ecology	Washington State Department of Ecology
EDL –	estimated detection limit
EPA –	U S Environmental Protection Agency
GC/MS –	gas chromatograph/mass spectrometer
IARC –	International Agency for Research on Cancer
ICP –	inductively coupled plasma
kg –	kilogram
K061 –	emission control dust/sludge from the primary production of steel in electric furnaces
mg –	milligram
meq –	milliequivalents
MTCA –	Model Toxics Control Act
ND –	non-detects
ngkg –	nanogram/kilogram
NT –	not tested
PCB –	polychlorinated biphenyl
PCDD/F –	polychlorinated dibenzodioxins and furans
PCP –	pentachlorophenol
ppb –	parts per billion
ppm –	parts per million
pptr –	parts per trillion
QA/QC –	Quality Assurance/Quality Control
RCRA –	Resource Conservation and Recovery Act
RPD –	relative percent difference
SWFAP –	Solid Waste and Financial Assistance Program
TCDD –	tetrachlorodibenzodioxin
TCDF –	tetrachlorodibenzofuran
TCLP –	toxicity characteristic leaching procedure
TEF –	toxicity equivalent factor
TEQ –	toxic equivalent
TOC –	total organic carbon
USDA –	U S Department of Agriculture
WAC –	Washington Administrative Code

Atomic Symbols for Metals

Arsenic	As
Barium	Ba
Cadmium	Cd
Chromium	Cr
Copper	Cu
Lead	Pb
Mercury	Hg
Nickel	Ni
Selenium	Se
Silver	Ag
Zinc	Zn

Abstract

This report is the result of Washington State Executive Request 1998 legislation (SSB 6474), *The Fertilizer Regulation Act,* mandating a study of dioxins in soils, soil amendments, and fertilizers. The Washington State Department of Ecology, with support from an EPA grant, expanded this study to include metals in fertilizers and soils.

The Department of Ecology, in cooperation with the Department of Agriculture and the Department of Health, conducted studies to (1) quantify metals and dioxins in fertilizer products, (2) determine if certain metals have accumulated in agricultural soils of the Columbia Basin, and (3) provide an initial assessment of typical concentrations of dioxins in statewide soils.

Seven fertilizer products failed state Toxicity Characteristic Leaching Procedure tests for cadmium. Five products are suspected to be derived from materials considered hazardous waste under Washington State regulations, but three of these appear to be exempt from regulation because the source material is steel mill flue dust. A few fertilizer products were found to contain relatively high levels of dioxin, but most products were very low in dioxin.

The data indicate that average metal concentrations in the soils sampled are higher in agricultural samples than background samples. However, only cadmium and zinc concentrations are statistically different. The metal concentrations found in this study do not indicate any increased risks to human health and the environment, but do indicate the need for periodic monitoring.

All soil samples had detectable levels of dioxins. The levels of dioxins detected in Washington State are comparable to other parts of the world.

Recommendations for future action are included in the report.

Executive Summary

This report is a result of Washington State Executive Request 1998 legislation (SSB 6474), *The Fertilizer Regulation Act,* mandating a study of dioxins in soils, soil amendments, and fertilizers. The Washington State Department of Ecology (Ecology), with support from an EPA grant, expanded this study to include metals in fertilizers and soils.

Some metals provide necessary micronutrients for plants and are constituents of some fertilizers, but metals can also be potentially hazardous tag-along contaminants of fertilizers. Heavy metals have been quantified in a number of fertilizers used in Washington State. *The Fertilizer Regulation Act* put Washington State Standards for metals in fertilizers into place.

Polychlorinated dibenzo-p-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs), together referred to as "dioxins," also pose a potential threat to human health and the environment as tag-along contaminants. Dioxins are found in some fertilizers and soil amendments, including fertilizers made from the recycling of industrial wastes used for their micronutrient value. Currently there are no standards for dioxins in fertilizers.

Objectives of the studies included:

- Quantify concentrations of metals and dioxins in fertilizers products
- Determine if certain metals have accumulated in agricultural soils of the Columbia Basin
- Provide an initial assessment of typical concentrations of dioxins in Washington soils
- To satisfy these objectives, Ecology, in cooperation with the state Department of Agriculture and the Department of Health, conducted studies to:
- Randomly sample fertilizer products to determine metal and dioxin concentrations
- Analyze metal concentrations of agricultural and non-agricultural soils in the Columbia Basin, and compare results with other state soil studies
- Sample soils in open, forest, and urban areas to determine if dioxins occur in these areas and, if so, at what levels

The Studies

1. Metals and Dioxins in Fertilizer Products

Fifty-one fertilizer products, including bulk agricultural fertilizers, home-use fertilizers, agricultural micronutrient products, and a soil amendment were sampled for eight heavy metals and 17 types of dioxins. The greatest number of samples analyzed was of home-use fertilizers, which includes a large variety of products.

Two bulk/packaged agricultural fertilizers, one agricultural micronutrient product, and four home-use fertilizers failed state Dangerous Waste Regulations (Toxicity Characteristic Leaching Procedure) tests for cadmium. Concentrations of cadmium in screening tests range from 1.5 to 2.5 parts per million (ppm), compared to the criterion of 1.0 ppm. Five products are suspected to be derived from materials considered hazardous waste under Washington State regulations. However, three of these products appear to be exempt from hazardous waste regulation, because they may be derived from steel mill flue dust which is exempt when used in fertilizer products. Ecology is concerned about fertilizers that fail the dangerous waste test and is evaluating these products.

Most fertilizers had non-detectable or extremely low levels of dioxin, with 70% having less than one-tenth of one part per trillion (pptr) toxic equivalents (TEQs). Three fertilizer products contained relatively high levels of dioxin. Two fertilizers had greater than 140 pptr, and one product exceeded 50 pptr. These three products are believed to contain steel mill flue dust, and had higher TEQs than any of the TEQs found in the Dioxins in Soils study.

2. Metals in Soils

Thirty-three sites were sampled in the Columbia Basin. Twenty samples came from agricultural soils and 13 from non-agricultural soils. The study area included portions of Adams and Grant counties within the Columbia Basin Irrigation Project where historical agricultural practices could be documented. Fields with historical use of biosolids (sewage sludge) and/or lead arsenate pesticides were excluded from this study.

The data indicate that, except for mercury, average metal concentrations in the soils sampled are higher in agricultural samples than background samples; however, only cadmium and zinc concentrations showed a statistically significant increase in agricultural samples when compared to background samples. Cadmium levels do not appear to pose a problem in plant uptake, due to the ratio of zinc to cadmium which favors zinc uptake before cadmium. The concentration of metals in soils tested in this study are at the low end of the range for Washington State soils, and do not indicate any increased risks to human health and the environment. The metal concentrations do indicate a need to periodically monitor soils to ensure levels do not become a concern.

3. Dioxins in Soils

Thirty soil samples were taken from urban, open, and forested lands and tested for 17 types of dioxins. All soil samples, including samples from remote wilderness areas, had detectable levels of dioxins. The data support other studies that dioxins are ubiquitous in the environment due, in large part, to aerial deposition. A comparison of results between the east and west side of the state, excluding urban samples, finds that sites sampled in eastern Washington tend to have lower levels of dioxin in soils.

Dioxin concentrations ranged from 0.033 to 19.5 pptr TEQ. Sample sites in the city of Tacoma had the two highest dioxin concentrations (9.5, 19.5 TEQ). The range of concentrations from urban areas was greater than those from the other two land use areas. Forest soils appear to have concentrations greater than soils from open areas. Concentrations of dioxins detected in Washington State soils are comparable to other parts of the world.

Recommendations

Future work necessary to satisfy *The Fertilizer Regulation Act* includes a survey of dioxins in agricultural soils. It is anticipated this study will occur in the spring of 1999.

Numerous recommendations are made in this report, including:

- Work cooperatively with the state Department of Agriculture to implement *The Fertilizer Regulation Act* by on-going review and sampling of fertilizer products
- Review existing information about dioxins in biosolids to provide the agencies with guidance for action in this matter
- Commit to a regulatory process to eliminate the steel mill flue dust exemption in Washington State Dangerous Waste Regulations
- Re-evaluate the wood ash exemption in the Washington State Dangerous Waste Regulations.
- Work with stakeholders to develop a strategy to minimize metals and dioxins in fertilizer products

This report also presents policy options for regulating toxic substances in fertilizer products.

Value of this Report

These studies will give fertilizer product manufacturers additional information that Ecology hopes the manufacturers will use to make cleaner products.

Farmers and home gardeners will have additional information to make informed decisions about the fertilizer products they purchase.

Information from these studies will assist Ecology in its effort to eliminate releases of cadmium, mercury, and dioxin - all bioaccumulative chemicals of concern - in the environment.

Introduction

Background

Concerns have been raised about whether metals and dioxins associated with waste-derived fertilizers and soil amendments pose a threat to human health and the environment. In response to these concerns, the Washington State Department of Agriculture and the Washington State Department of Ecology (Ecology) tested 55 fertilizers for 24 metals during the spring of 1997 This screening study (Bowhay et al., 1997) concluded that the concentration of metals in most fertilizers tested met the Canadian standards for metals in fertilizers Additionally, a few waste-derived fertilizers suspected to contain dioxins and furans were tested for dioxins All of the waste-derived products tested, except elemental lime, contained dioxins

As of 1997, no federal or state standards existed to regulate the level of contaminants in most fertilizer products During late 1997 and early 1998, information from the state screening study was given to the Fertilizer Advisory Workgroup, the Legislature, and the Governor's Office In early 1998, the Legislature passed Executive Request legislation (SSB 6474), *The Fertilizer Regulation Act.* The Act (1) adopted state standards, based on the Canadian standards, for metals in all fertilizers, (2) increased Ecology regulatory oversight of waste-derived fertilizers and soil amendments, and (3) mandated labeling on the metals content for all fertilizers The Act also mandated a study of additional metals and dioxins in soils, soil amendments, and fertilizers as well as a plant-uptake of metals study, initiated this year. These studies will give the Legislature and others information to determine if further fertilizer regulation is needed

Study Objectives and Summaries

Fertilizers and soil amendments from natural, manufactured, and industrial by-product sources can contain "tag-along" substances that have little or no nutrient value Additionally, some materials classified as hazardous and solid wastes under existing Ecology regulations are recycled as ingredients in fertilizers and soil amendments

- Certain elements (e g , zinc), as constituents of fertilizers, are recognized as necessary nutrients required for plant life Some metals can be potentially hazardous tag-along contaminants in fertilizers Heavy metals have been quantified in a number of fertilizers used in Washington State (Bowhay et al., 1997) These metals are naturally occurring elements, but fertilizer use, over long periods of time, may increase the metal concentrations in agricultural soils

- Polychlorinated dibenzo-p-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs), together referred to as “dioxins” in this report, also pose a potential threat to human health and the environment as tag-along contaminants and have been quantified in some waste-derived fertilizers used in Washington State (Bowhay et al , 1997)

The legislation signed by Governor Locke in 1998 directs Ecology, in cooperation with the state Department of Agriculture and the state Department of Health, to "undertake a study to determine if dioxins occur in fertilizers, soils amendments and soils and, if so, at what levels " (Washington State Legislature, 1998). From May through October 1998, Ecology conducted studies to investigate (1) metals and dioxins in fertilizers and soil amendments, (2) metals in soils, and (3) dioxins in non-agricultural soils While some conclusions may be drawn from these studies, their limited scope qualifies them as screening studies They will help direct further research, if needed, to provide information for possible regulation of these substances

This legislation also directed the state Department of Agriculture, which contracted with Washington State University, to conduct a longer crop-uptake study That study, initiated in the fall of 1998, will evaluate the uptake of metals in certain crops in relationship to the new Washington fertilizer standards This report does not contain the crop-uptake of metals study

1. Metals and Dioxins in Fertilizer

The objective of this study is to quantify metals and dioxins in (1) bulk agricultural fertilizers, (2) home-use fertilizers, (3) micronutrients, and (4) soil amendments This report will use the term "fertilizer products" to refer to all products sampled, which includes one soil amendment Ecology randomly sampled and analyzed bulk agricultural and home-use fertilizers, as well as soil amendments, to determine dioxin concentrations Zinc micronutrient fertilizers were also analyzed to determine heavy metal concentrations, as part of an U S Environmental Protection Agency (EPA) grant Metals analyzed were arsenic, barium, cadmium, chromium, lead, mercury, selenium, and silver These eight metals are a subset of the metals analyzed in the 1997 study (Bowhay et al , 1997) and are the metals of greatest concern The fertilizers were also analyzed using the toxicity characteristic leaching procedure (TCLP) which is used in designating hazardous and dangerous wastes

Results from this study will help assess the effectiveness of Ecology's waste-derived and micronutrient fertilizer screening criteria (Washington State Register, 1998). As required by *The Fertilizer Regulation Act*, Ecology developed the screening criteria to review fertilizer registration applications. This review will ensure fertilizers meet the applicable federal hazardous waste and state dangerous waste regulations

2. Metals in Soils

The objective of this study is to determine if certain metals have accumulated in agricultural soils of the Columbia Basin. Ecology analyzed metal concentrations of agricultural and non-agricultural soils in the Columbia Basin and compared these results with results from two other state soil studies Metals analyzed were arsenic, cadmium, copper, lead, mercury, nickel, and zinc These seven metals were selected for comparison with other applicable soils studies "Agricultural" land is defined as land in active agricultural production "Non-agricultural" land is land that has never been farmed, tilled, or grazed

Comparisons are made of metal concentrations of agricultural and non-agricultural soils in the Columbia Basin Irrigation Project District

3. Dioxins in Soils

The objective of this study is to provide an initial assessment of typical concentrations of dioxins in Washington State soils. Ecology sampled soils in open, forest, and urban areas to determine if dioxins occur in these areas and, if so, at what levels. The original intent of a portion of this study included sampling and analyzing agricultural soils for dioxins However, due to difficulties in randomly selecting agricultural sampling sites and not being able to guarantee confidentiality to land owners, agricultural soil collection and analysis was not conducted this year Ecology is currently exploring strategies to maintain randomness and assure confidentiality The agency hopes to conduct agricultural soil sampling in mid-1999 When this sampling is completed and results analyzed, an addendum to this report will be published

With this information, Ecology will be better able to develop strategies for restricting substances containing high dioxin levels from being added to soils. Possible sources of dioxins in the soils sampled will not be addressed by this study

Background Information on Metals and TCLP

Metals

Although elemental metals occur naturally, Ecology is concerned about metals that may unintentionally be distributed to the environment in relatively high concentrations

Arsenic, although naturally occurring in Washington State, can be harmful to human health and the environment in excessive concentrations No evidence exists that arsenic is beneficial to humans Arsenic was used for many years in pesticide products, including use as a wood preservative, and is currently used to increase hardening and heat resistance in glassware and ceramics Ore deposits, mining activities, and industrial and manufacturing processes are also sources of arsenic. Chronic inhalation of arsenic causes lung cancer in smelter and pesticide workers Cancers of the bladder, lung, liver, kidneys, and skin have been associated with ingestion of arsenic (DOH, 1996a)

No evidence exists that **cadmium** is biologically essential or beneficial In sufficient concentration, it is toxic to all forms of life including higher plants, animals, and humans Cadmium is a relatively rare metal, usually present in small amounts in zinc ores, and it is commercially obtained as an industrial by-product of the production of zinc, copper, and lead Background levels of cadmium in crops and other plants are usually <1 0 mg/kg (ppm) Little is known about the cadmium concentrations resulting in reduced plant yields, however, plants growing in cadmium-contaminated soils contain abnormally high residues that may be detrimental to plant growth, as well as to animal and human consumers (Eisler 1985) Exposure to low levels of cadmium over a long period of time can cause severe and irreversible damage to the kidneys (DOH, 1996b)

Mercury has been used by man for at least 2300 years Industrial uses currently include use as an agricultural fungicide, as a slime control agent in the pulp and paper industry, and in the production of plastics Mercury and its compounds have no known biological function, and the presence of the metal in the cells of living organisms is undesirable and potentially hazardous Mercury, as with cadmium, can be bioconcentrated in organisms and biomagnified through food chains Mercury is a mutagen, teratogen, carcinogen, and can be lethal to embryos (Eisler, 1987)

Lead is a naturally occurring metal found in small amounts in the earth's crust Most elevated concentrations of lead in the environment results from human activity, and it is found throughout the environment (e.g , in air, drinking water, rivers, lakes, oceans, dust, and soil). Lead is very persistent in soils and may remain in soil for years It can also bioaccumulate in food chains Excessive lead can cause anemia, severe damage to the brain and kidneys, and severe reproductive effects Children are more sensitive to lead than adults; their lower body weights and incomplete neurological development increase their sensitivity to lead (ATSDR, 1997)

Zinc is one of the most common elements in the earth's crust. Zinc is widely used in industry. It is used in several alloys, dry cell batteries, wood preserving, pharmaceutical products, and is a necessary plant nutrient in fertilizers When zinc is deposited on land, it is usually bound to the soil and does not dissolve in water Zinc is present in most foods eaten every day, the average daily dietary intake in the United States ranges from 7 to 16 3 milligrams (mg) Recommended Dietary Allowances are 15 mg/day for men and 12 mg/day for women (ASTDR, 1994) Ingestion of large doses (100-150 mg) of zinc by humans over a short period of time can cause stomach cramps and vomiting Prolonged consumption of excess zinc may damage the pancreas, as well as cause irritability, loss of appetite, nausea, vomiting, and anemia It can also interfere with the ability of the body to absorb and use other minerals, such as copper and iron (DOH, 1996c)

TCLP

The Toxicity Characteristic Leaching Procedure (TCLP) is an extraction procedure developed to model the leaching a waste undergoes if disposed in a municipal solid waste landfill Decomposing municipal solid waste produces a slightly acidic leachate that can extract hazardous constituents from the waste (Ecology, 1998). This procedure is used to determine the extent to which certain contaminants would become available to migrate in the leachate The TCLP procedure is used to determine if a solid waste is also a hazardous waste (WAC 173-303, Dangerous Waste Regulations), and has recently been incorporated into Ecology's Review of Waste-Derived and Micronutrient Fertilizers If a waste-derived fertilizer fails certain limits for TCLP (Appendix 1-I), the state Department of Agriculture will not recommend the fertilizer for registration

Background Information on Dioxins

The following background information is a summary from the *Washington State Dioxin Source Assessment* (Yake et al , 1998).

Dioxins are unintended by-products formed during combustion of organic compounds in the presence of chloride, incineration of municipal and hospital waste, and chlorine bleaching of wood pulp (Alcock and Jones, 1996, Birnbaum, 1994, Rappe, 1984). The production of certain chlorinated organic products also produces dioxins; they are contaminants in certain chlorinated organic products (e g , pentachlorophenol [PCP], a wood preservative). Dioxins have no commercial or domestic applications and are not intentionally produced, except for small quantities used in research (ATSDR, 1989, Federal Register, 1997)

Polychlorinated dioxins and furans are persistent environmental pollutants that accumulate, primarily through food chains, in the tissues of animals, including humans The International Agency for Research on Cancer has concluded that 2,3,7,8-TCDD is a "known human carcinogen" (IARC, 1997) Exposures to PCDDs and PCDFs are associated with enzyme induction, chloracne, immunotoxicity, developmental toxicity, and cancer in both animals and humans (Birnbaum, 1994)

Recent concern about the effects of dioxins on organisms has increasingly focused on endocrine disruption and reproductive impairment (EPA, 1997) The EPA states " 2,3,7,8-TCDD is one of the most, if not the most, potent reproductive/developmental toxicants known" and "studies in various animal species have also demonstrated that the immune system is a target for toxicity of 2,3,7,8-TCDD" (Federal Register, 1997)

Dioxins are found in some fertilizers and soil amendments made from the recycling of industrial wastes that are used for their micronutrient or liming properties (e.g , zinc) (Bowhay et al , 1997) Currently no standards exist for dioxins in fertilizers that can be used to make clear, defensible decisions about which fertilizers and soil amendments should be used on croplands or pasturelands

What are dioxin TEFs and TEQs?

Dioxins and related compounds usually occur in complex mixtures Of the 210 forms or congeners of polychlorinated dibenzo-*p*-dioxins and polychlorinated dibenzofurans, 17 dioxin and furan congeners are considered toxic The congeners are identified by the number and location of chlorine atoms on the molecule The most toxic of these congeners have chlorine atoms at four specific sites (the 2,3,7, and 8 positions) The most toxic dioxin congener is 2,3,7,8-tetrachloro dibenzo-*p*-dioxin (2,3,7,8-TCDD) Other similar dioxins have been assigned toxicity values relative to it (Birnbaum, 1994) In this report the terms "dioxin" and "dioxins" refer to all 2,3,7,8-substituted PCDDs and PCDFs unless otherwise noted See Figure 1 for the generalized chemical structure and numbering of dioxin

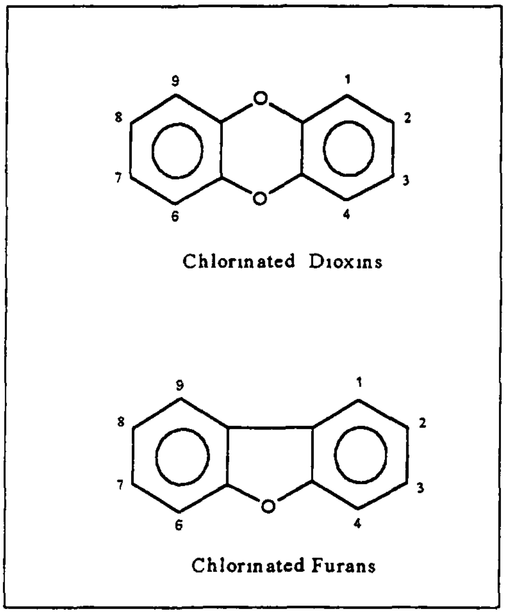

Figure 1. Chemical structure of dioxins/furans.

The toxicity values for these compounds relative to 2,3,7,8-TCDD are called toxicity equivalency factors (TEFs). 2,3,7,8-TCDD is assigned a TEF of 1, and the others are assigned values less than 1 TEFs are used to express a total toxicity of dioxins when the concentration of each congener is multiplied by its TEF and all the products are added up.

The total toxicity of a material containing several dioxin congeners is reported as toxicity equivalents (TEQs)[1] An example of this calculation is shown in the footnote below See Appendix 1 for further information

[1] The calculation of TEQ for a media sample containing 5 pptr 2,3,7,8-TCDD and 23 pptr 2,3,7,8,-TCDF (considered 1/10 as toxic as TCDD, it has a TEF of 0 1) is [5+(0 1 x 23)] = 7 3 pptr TEQ (Serdar et al , 1991)

1. Metals and Dioxins in Fertilizer Products

Purpose

The objective of this study is to quantify metals and dioxins in (1) bulk agricultural fertilizers, (2) home-use fertilizers, (3) micronutrients, and (4) soil amendments.

In this report, the term "fertilizer products" refers to all four of the above substances

Results from this study will help assess the effectiveness of state screening criteria for waste-derived fertilizers and micronutrients Ecology randomly sampled and analyzed bulk agricultural and home-use fertilizers, micronutrients, and a soil amendment to determine their dioxin concentrations. The samples were also analyzed to determine heavy metal concentrations, as part of an EPA grant. Metals analyses of fertilizer products included total metal concentrations and leaching metal concentrations

Study Design

To determine which products to sample, random samples were drawn from among products registered for use in Washington State in three fertilizer groups bulk/packaged agricultural fertilizers, agricultural products with micronutrients, and home-use fertilizer products Home-use fertilizer products were randomized using a random number generator, and the first 31 products were selected Bulk/packaged agricultural fertilizers were categorized by constituent type, randomized, and the first few products per category were selected. Micronutrients were selected at random, with available products substituting for those that could not be obtained

Fertilizers are defined under *The Fertilizer Regulation Act* (SSB 6474) as containing commercially valuable concentrations of nitrogen, phosphoric acid, available phosphorus, potash, calcium, magnesium, or sulfur Micronutrients are boron, chlorine, cobalt, copper, iron, manganese, molybdenum, sodium, and zinc A non-nutritive soil amendment, Ponderay Newsprint Fiberay SC, was also sampled.

The greatest number of samples were of home-use fertilizers, which included a large variety of products There are considerably fewer agricultural fertilizers in use This study focused on zinc as an agricultural micronutrient, because zinc micronutrients were associated with relatively high levels of dioxin in some products tested by Ecology in 1997 (Golding, 1997) Agricultural products were sampled at distributors of agricultural chemicals Home-use fertilizer products were obtained in the form sold to consumers (i e , "off-the-shelf") Not all home-use fertilizers registered in Washington State are sold in the state, and some selected home-use fertilizer samples could not be obtained In these cases, a commonly found product with similar constituents and usage was substituted A list of fertilizers, soil amendments, and micronutrients sampled appear in Appendix 1-A

Fifty-nine samples (51 different products) were analyzed for eight heavy metals and dioxins. This number included two samples each of Cozinco zinc micronutrient and Frit F-503G obtained independently from different suppliers, as well as six duplicate samples of other fertilizer products. Duplicates are samples taken from a single mixed sample in order to determine replicability of results

Sampling Procedures

Samples were collected based on procedures described in the Washington State Department of Agriculture Investigator's Manual, Pesticide Management Division (WSDA, 1991) The following is a description of the modified sampling procedure

Samples were taken using organic-free, laboratory-cleaned sample jars and pre-cleaned stainless steel ladles Bulk solid samples were collected as grab-composite samples consisting of ten grab subsamples from discrete parts of a product being sampled Packaged solid samples were obtained as grab samples by filling a mixing bowl approximately 2/3 full. The subsamples and grab samples were combined in a cleaned, four-quart stainless steel mixing bowl, mixed with the sampling ladle, and split into Ecology and facility sample containers Sample jars were ultra-clean with Teflon lids Bulk liquid was sampled by flushing a sample port and collecting the sample as a grab sample directly into a clean glass sample bottle The cleaning regimen is listed in Appendix 1-B Because of the special cleaning requirements for PCDD/PCDF sampling, cleaned stainless steel ladles were used in lieu of the triers specified in the Washington State Department of Agriculture Investigator's Manual

Sample jars were labeled with tags and placed in plastic bags. All samples were stored in a cooler and maintained at a temperature of 4°C until analysis Chain-of-custody procedures followed the Manchester Environmental Laboratory Lab Users Manual (Ecology, 1994) The samples for this project were delivered to the Manchester Laboratory by Ecology staff

Analytical Procedures

Analysis of total metals was carried out by either graphite furnace atomic absorption or inductively coupled plasma (ICP) optical emission spectroscopy, depending on analyte level and matrix interference EPA SW-846 Method 6010 was used for ICP For graphite furnace analyses, SW-846 Methods 7421 (lead), 7131 (cadmium), 7740 (selenium), 7761 (silver), and 7060 (arsenic) were used Mercury analysis was carried out by cold vapor atomic absorbance, SW-846, Method 7471 Toxicity Characteristic Leaching Procedure (TCLP) analyses were carried out in accordance with SW-846, Method 1311. Method references appear in Appendix 1-C

Analysis of the 2,3,7,8-substituted PCDD/PCDF congeners (forms of dioxins and furans) was conducted at MAXIM Technologies Inc /Pace Analytical, using high resolution GC/MS EPA Method 8290, with enhancements derived from Method 1613B

Detection limits for dioxins varied depending upon the physical and chemical characteristics of the samples, with a target detection limit of 0 1 pptr (parts per trillion) EPA Method 8290, Section 7 9 5 specifies the sample specific Estimated Detection Limit (EDL) as the concentration of a given analyte required to produce a signal with a peak height of at least 2 5 times the background signal level Not all the analyses for congeners were responsive enough to provide EDLs down to 0 1 pptr

Data Quality

Established laboratory quality control procedures were adequate to estimate laboratory precision and accuracy for this project. Laboratory quality control tests were done on each set of 20 or fewer samples and consisted of blanks, duplicate samples, and spiked samples Laboratory quality control and procedures are discussed in the Manchester Environmental Laboratory Lab Users Manual (Ecology, 1994a).

Metals

Metals results can be used without qualification except in those cases with low spike recoveries and poor duplicate precision Qualifiers appear in the data tables included in this report A discussion of QA/QC for metals appears in Appendix 1-D

A comparison of multiple sample metals results as well as duplicate sample results appears in Appendix 1-E Duplicate sample results were in close agreement The average relative percent difference (RPD) for pairs of detected metals was 23% and the range of RPDs was relatively tight, indicating consistency in sampling and analysis. RPD, a measure of precision, is the ratio of the difference and the mean of the results expressed as a percentage A low RPD indicates high precision

Dioxin

Quality Assurance/Quality Control (QA/QC) measures indicate that the dioxin results are reliable One sample (328132) exceeded the allowable 30-day holding period by one day, but holding time is not considered critical for dioxin Calibration standards, internal standard recoveries, ion abundance ratios, and matrix spike/matrix spike duplicates were acceptable A more complete discussion of QA/QC appears in Appendix 1-C

In the fall of 1997, when Ecology sampled several waste-derived fertilizer products, dolomite was sampled to serve as a blank sample No 2,3,7,8-substituted PCDD/PCDF congeners were detected in the dolomite sample, yielding a TEQ of 0 for the case where undetected congeners are assumed to have a dioxin concentration of 0 (minimum value) The TEQ (total equivalent toxicity) was 0 84 pptr for the "worst case" (maximum value) where the calculation of TEQ is made, assuming all undetected congeners were present at the detect limit (i e , non-detects set to the detection limit [ND=DL]) See Appendix 1 for a discussion of TEQ calculations Of the 51 fertilizer products sampled in this study, no 2,3,7,8-substituted PCCD/PCDF congeners were detected in 13 of the fertilizer products These results indicate that the sampling and analysis techniques employed were capable of measuring small concentrations of dioxin without significant field or laboratory contamination

Field quality assurance for this project consisted of six duplicate split samples. The differences in duplicate sample results reflect combined sampling and laboratory variability. All duplicate sample TEQs (for ND=0) were within 0 34 pptr (Appendix 1-F) The RPD between the duplicate split sample results was not calculated, because RPDs are not meaningful when results approach zero Because the TEQs of duplicated samples were low, conclusions cannot be drawn concerning the replicability of samples with larger TEQs

Results and Discussion

Total Metals

Metals analyses results are shown in Appendix 1-G Each value represents the result of a single composite sample of a product.

Table 1-1 shows the fertilizer products with the five highest total metal concentrations for each metal tested and the products' metal concentrations

From Table 1-1 it can be seen that the fertilizer products with the highest concentrations of metals were NuLife All-Purpose Trace Elements (75 2 mg/kg-dw arsenic), Fort James Nutri Lime (543 mg/kg-dw barium), Agrium Ammonium Phosphate Sulfate (160 mg/kg-dw cadmium), McLendon Weed and Feed 15-5-5 (5,060 mg/kg-dw chromium), Frit F-420G (9,490 mg/kg-dw lead), Frit F-503G (10 06 mg/kg-dw mercury), Frit F-420G (5.60 mg/kg-dw selenium), and Frit F-420G (20 6 mg/kg-dw silver)

The appearance of a product in this list does not necessarily indicate that the concentrations are of concern. The fertilizer products that were sampled were in the channels of trade prior to the 1998 registration. As a result, some of these products may no longer be available or may have been reformulated

Ecology is currently in the process of obtaining application rate information for the products that have the higher concentrations of arsenic, cadmium, lead, mercury, and selenium in order to assess whether these products fail the Washington Standards adopted in RCW 15 54 800 These standards are the maximum annual metal additions to soil allowed in Washington. It should be noted that the metals analyzed in this study are not in all cases those used by the Washington State Department of Agriculture to regulate heavy metals in fertilizers

Two of the five fertilizer products with the highest cadmium concentrations are phosphate fertilizers: Agrium ammonium phosphate sulfate and UAP 0-45-0. As noted in last year's screening survey (Bowhay, et al, 1997), phosphate fertilizers had relatively high amounts of product applied per acre (loading rates) There is much more phosphate fertilizer applied in the

Table 1-1. Five highest rank ordered total metal concentrations in fertilizer products - 1998 sampling results.

Arsenic (As)

Abbreviated Product Description	*mg/kg-dw*
NuLife All-Purpose Trace Elements	75.2
Frit F-420G *	40.1
Fort James Nutri Lime	28.5
Frit F-503G **	27.2
Whitney Farms Jersey Green Sand	11.4

Barium (Ba)

Abbreviated Product Description	*mg/kg-dw*
Fort James Nutri Lime	543
NuLife All-Purpose Trace Elements	205
Frit F-420G *	181
Osmocote Vegetable and Bedding	141
Frit F-503G Sample #2	130

Cadmium (Cd)

Abbreviated Product Description	*mg/kg-dw*
Agrium Ammonium Phosphate Sulfate	160
Frit F-420G *	135
UAP 0-45-0	106
Pace NuLife 10-20-20	89.3
Webfoot Rhododendron	70.4

Chromium (Cr)

Abbreviated Product Description	*mg/kg-dw*
McLendon Weed and Feed 15-5-5	5060
Frit F-420G *	1060
Webfoot Rhododendron	612 J
NuLife All-Purpose Trace Elements	417
UAP 0-45-0	378

Lead (Pb)

Abbreviated Product Description	*mg/kg-dw*
Frit F-420G *	9490
Gaia's Own Cottonseed Meal	2550
Frit F-503G **	2039
NuLife All-Purpose Trace Elements	1940
Hydro-Feed with Polyon 20-10-10	434

Mercury (Hg)

Abbreviated Product Description	*mg/kg-dw*
Frit F-503G **	10.06
Terosa Rose Food	1.13
Frit F-420G *	1.09
Pursell Sta-Green Nursery Special	0.652
Pursell Sta-Green Azalea	0.364

Selenium (Se)

Abbreviated Product Description	mg/kg-dw
Thrifty Pay-Less Tomato & Veg	5.71
Frit F-420G *	5.60
Terosa Rose Food	2.60
NuLife All-Purpose Trace Elements	1.90
Whitney Farms 100% Organic Citrus	1.1

Silver (Ag)

Abbreviated Product Description	mg/kg-dw
Frit F-420G *	20.6
Frit F-503G **	5.64
NuLife All-Purpose Trace Elements	5.28
Tech-Flo Zeta Zinc 22	3.20
Cozinco Sample #1	3.0

J - estimated value

*The sample of this material was collected in Oregon. The product is not registered or sold in Washington

** Frit F-503G values are averages from two independent samples state than micronutrient fertilizer. Cadmium was found to be elevated in agricultural soils in the Metals in Soils study (Chapter 2). The study found a significant increase in cadmium in the agricultural soils sampled, as opposed to background non-agricultural soil samples

TCLP Metals

The new fertilizer review process, beginning July 1, 1999, requires that Ecology

- Review waste-derived fertilizers and micronutrients to determine if products pass dangerous waste criteria
- Advise if these products should be registered by the Washington State Department of Agriculture for use as a fertilizer in Washington State (Washington State Register, 1998)

The dangerous waste criteria include the Toxicity Characteristic Leaching Procedure (TCLP) In this new review process, if a product fails TCLP it will not be recommended for registration TCLP testing was conducted on certain fertilizer products in order to help assess the effectiveness of waste-derived and micronutrient fertilizer screening criteria

In this study, products were selected for TCLP testing based on their total metal concentrations In accordance with Method 1311, all samples for which total metal concentrations equaled or exceeded 20 times the dangerous waste limit for the dangerous waste toxicity characteristic (WAC 173-3033-090) were tested using the TCLP The "20 times rule" does not apply to liquid samples, the liquid sample serving as the leaching extract directly The results of these tests are shown in Table 1-2

Dangerous waste limits for TCLP are shown in Appendix 1-H Seven of the 51 products tested in 1998 (Table 1-2) exceeded TCLP limits for cadmium These products were (1) Agrium Ammonium Phosphate Sulfate, (2) United Agri Products 0-45-0, (3) Frit F-503G, (4) Webfoot Rhododendron, Camelia, and Azalea Food, (5) NuLife 10-20-20, (6) NuLife Agro 10-15-10, and (7) Thrifty Payless Tomato and Vegetable Food (Table 1-3) Two of the seven were bulk or packaged agricultural fertilizers, one was an agricultural micronutrient, and four were home-use packaged fertilizer products

Ecology will not recommend waste-derived products failing the TCLP test for fertilizer registration by the Washington State Department of Agriculture To make that determination, additional information about the source of the ingredients in these products is needed As part of this study, Ecology is following up with the companies that manufacture these products to obtain that information

7

Table 1-2. TCLP metals test results of fertilizers - 1998 sampling results.

1998 Sampling Results

	As Mg/L	Ba mg/L	Cd mg/L	Cr mg/L		Pb mg/L		Hg mg/L	Se mg/L	Ag mg/L	Lab Log#
TCLP Limit	**5.0**	**100**	**1.0**	**5.0**		**5.0**		**0.2**	**1.0**	**5.0**	
Abbreviated Product Description											
Frit F-503G Sample #1				0 055		0 536		0 0007			318086
Frit F-420G			0 125	0 005	U	1 87					318087
Webfoot Rhododendron duplicate			**1.53**	2 49							**328126**
Agrium Ammonium Phosphate Sulfate			**2.25**	0 208							**328131**
Webfoot Rhododendron			**1.50**	2 30							**328140**
NuLife All-Purpose Trace Elements			0 907	0 008		0 111	J				328144
Terosa Rose Food			0 277	0 040		0 053	J				328146
Gaia's Own Cottonseed Meal						0 02	U				338183
Hydro-Feed with Polyon 20-10-10						0 02	U				338187
McLendon Weed and Feed 15-5-5				4 57							338190
Pace NuLife 10-20-20			**2.23**	0 258							**338194**
NuLife Agro 10-15-10			**1.36**	0 142							**338195**
NuLife Agro 10-15-10 duplicate			**1.04**	0 118							**338196**
Winter Green 15-10-25			0 032	0 075							338197
J R Simplot Best 6-20-20XB			0 903	0 171							338198
Thrifty Pay-Less Tomato and Veg.			**1.26**	0 101							**338205**
A H Hoffman Ace Tomato and Veg				**0.175**							348209
UAP 0-45-0			**2.16**	0 491							**348210**
Frit F-503G Sample #2			**2.52**	0 05	U						**348214**

bold - Value exceeds TCLP limit
U - Analyte was not detected at or above the reported result
J - Analyte was positively identified Associated numerical result is an estimate
UJ - Analyte was not detected at or above the reported estimated result

Table 1-3. Summary of fertilizer samples failing TCLP tests for cadmium.

Bulk/Packaged Agricultural Fertilizers

Lab Log#	*Sample ID*	TCLP *Conc.ppm*	*Product Name*
328131	AGRIUM	2.25	Agrium Ammonium Phosphate Sulfate
348210	UAP45	2.16	United Agri Products 0-45-0

Agricultural Micronutrients

Lab Log#	*Sample ID*	TCLP *Conc.ppm*	*Product Name*
348214	FTF503G	2 52	Frit F-503G

Home-Use Packaged Fertilizer Products

Lab Log#	*Sample ID*	TCLP *Conc.ppm*	*Product Name*
328126	WEBRHOD Dupe	1 53	Webfoot Rhododendron Food
328140	WEBRHOD	1.5	Webfoot Rhododendron Food
338194	NL102020	2 23	NuLife 10-20-20
338195	NL101510	1.36	NuLife 10-15-10
338196	NL101510 Dupe	1.04	NuLife 10-15-10
338205	PAYLESS	1 26	Thrifty Payless Tomato and Vegetable Food

Results of the multiple independent sampling of some fertilizer products are presented in Appendix 1-E

Comparison of Metals Results with the Findings of Other Sampling Events

Three of the 51 fertilizer products sampled in July-August 1998 for this study were previously sampled in January 1998 (Kelly Green Fresh Fish Fertilizer, Cozinco and QC 30% zinc) Although these products have elevated levels of cadmium, chromium, and lead, they do not exceed the TLCP limit by 20 times, so no TCLP testing was warranted Results were close for both sampling dates, with an average relative percent difference (RPD) for detected pairs of metals of 32%. Appendix 1-I shows a comparison between the metals results Appendix 1-J shows the January 1998 results Appendix 1-K lists the materials or product names and manufacturers of the products shown in Appendix 1-J

Metals results obtained in a 1997 sampling survey of several waste-derived fertilizer product sources are shown in Appendix 1-L Four of the products sampled in the fall of 1997 (Golding, 1997) were also sampled earlier in 1997 (Bowhay et al , 1997) In that study, a Holnam cement kiln dust sample was found to have 150 mg/kg-dw of total lead In the fall 1997 study, Holnam cement kiln dust had 230 mg/kg-dw of total lead TCLP tests of Holnam cement kiln dust have shown no exceedance of TCLP limits (Stone, 1998)

Bay Zinc Company, Inc has produced several zinc micronutrient products from several sources of zinc-containing material A comparison of metals results shows that Bay Zinc 18% Blu-Min micronutrient samples obtained for the 1997 metals screening study and the 1997 dioxin study had close results, with an average RPD for paired detected metals of 9% 18% Blu-Min was derived from K061 steel mill furnace dust. When K061 is recycled into zinc micronutrient fertilizer, it is exempt from dangerous waste requirements (WAC 173-303-071) At the time of the 1997 dioxin study in the fall of 1997, samples from 188 tons of Bay Zinc LHM failed TCLP for cadmium The material has since been approved by Canada for importation as a fertilizer and has been exported (Granberg, 1998)

Dioxin

A summary of TEQs for fertilizers sampled in 1998 appears in Appendix 1-M, and TEQ calculations are found in Appendices 1-N and 1-O In this study, unless otherwise noted, TEQs are calculated based on non-detects set to 0 (ND=0) When a compound being analyzed for is not detected, the result is termed a "non-detect" and the true sample concentration of that compound is not known, falling somewhere between zero and the detection limit (DL) of the analysis The three methods of calculating the TEQs that appear in Appendix 1-M are explained in Appendix 1 Each value represents the results of a single composite sample of a product. See Appendix 1-P for a summary of results for fertilizer products and micronutrients sampled in 1997 (Golding, 1997).

A few fertilizer products were found to contain relatively high levels of dioxin. Three fertilizer products had TEQs of greater than 50 pptr NuLife All-Purpose Trace Elements had a TEQ of 53.7 pptr Two of the products, Frit F-420G (287 pptr) and Frit F-503G (145 pptr), had TEQs of greater than 100 pptr (Figure 1-1; Appendix 1-M) Frit F-503G had less than 100 pptr in a separate sample (26 8 pptr) All other fertilizer products had TEQs of less than 10 pptr The Fnt products are micronutrients beheved to be derived from steel mill flue dust (Bowhay, 1998) The Cozinco micronutrient product and other zinc micronutnent products had low TEQs compared with the Frit sample results The Cozinco product is derived from galvanizing waste (Bowhay, 1998)

The results of analyses for two independently collected samples of the Cozinco 35 50% zinc micronutrient from different suppliers were close, with TEQs within 0 1 pptr. (Appendix 1-F) This shows good agreement between the two samples and their analyses The results for the two independently collected Frit F-503G samples diverged by a factor of 5. Metals results for these two samples also varied considerably, indicating that the product as sampled was not consistent with respect to the metals tested

Figures 1-1 and 1-2 show dioxin TEQs for individual product samples, rank-ordered by TEQ Figure 1-3 shows a frequency dıstribution of TEQs for dioxin results of the fertilizer products tested Figure 1-3 and Appendix 1-M show that 36 of the 51 products sampled (71%) had TEQs of less than 0 1 pptr (there are 37 total samples but two samples of the Cozinco micronutrient product show as less than 0 1 pptr)

In 1997, seven waste-derived fertilizer products were tested for dioxin One of these products, 18% Blu-Min micronutrient, had a TEQ of 342 pptr (Figure 1-2; Appendix 1-P) The product was marketed by Bay Zinc Company, Inc and was derived from steel mill flue dust, K061. Fort James NutriLime was tested in 1997 and again in 1998 NutriLime is fly ash from a Fort James hog fuel boiler The TEQ for the Fort James NutriLime sampled in the October 1997 (35 4 pptr) was greater than the TEQ for the August 1998 sample (7.35 pptr) by almost a factor of 5 This may be the result of differences in hog fuel boiler fuel or operating conditions (Young, 1998).

The selection process differed between the 1997 and 1998 studies The products tested in 1997, with the exception of dolomite, were selected because they were waste-derived products associated with known or reported sources of dioxin, whereas the products tested in 1998 were randomly selected from all registered fertilizers.

Comparison of Dioxin Results with the Findings of Other Studies

A literature review found no studies of dioxin levels in fertilizers There are no applicable standards for dioxin in fertilizers at this time Fertilizer dioxin levels can be compared with the results of the Dioxins in Soils study (Chapter 3)

Most fertilizer products tested in 1998 had low dioxin toxicity levels, with 71% of the products sampled having TEQs of less than 0 1 pptr This can be compared with the soils dioxin study that represented typical non-agricultural soils throughout Washington state (Chapter 3) That study found only 17% of soil samples with a TEQ of less than 0 1pptr The reason for the higher soil levels of dioxin may be that soil is more subject to atmospheric deposition of dioxin than fertilizers (Czuczwa et al , 1984, Czuczwa and Hites, 1986, Creaser et al., 1989; Rotard et al , 1994)

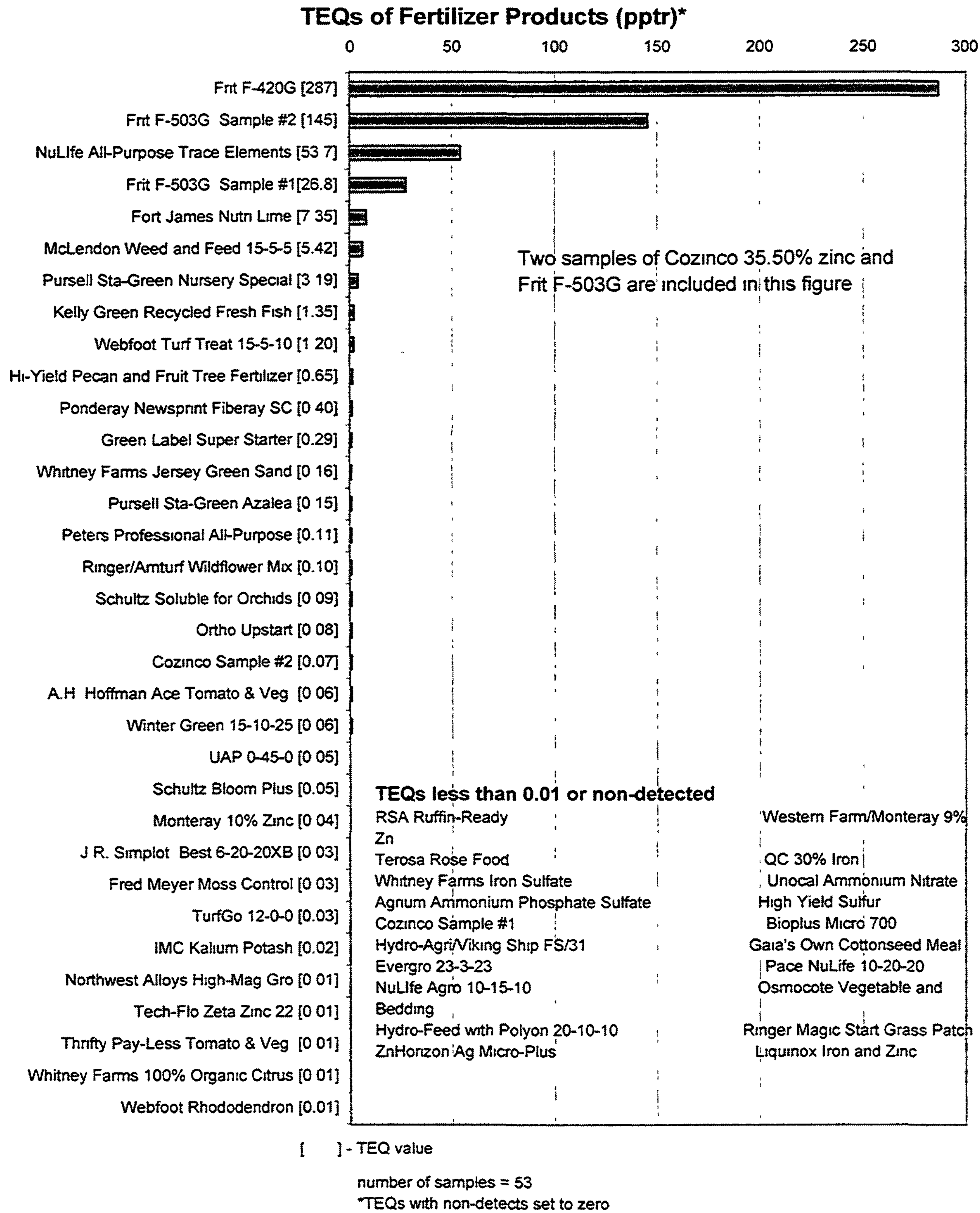

Figure 1-1. Rank-ordered TEQs in fertilizer products -1998 sampling results.

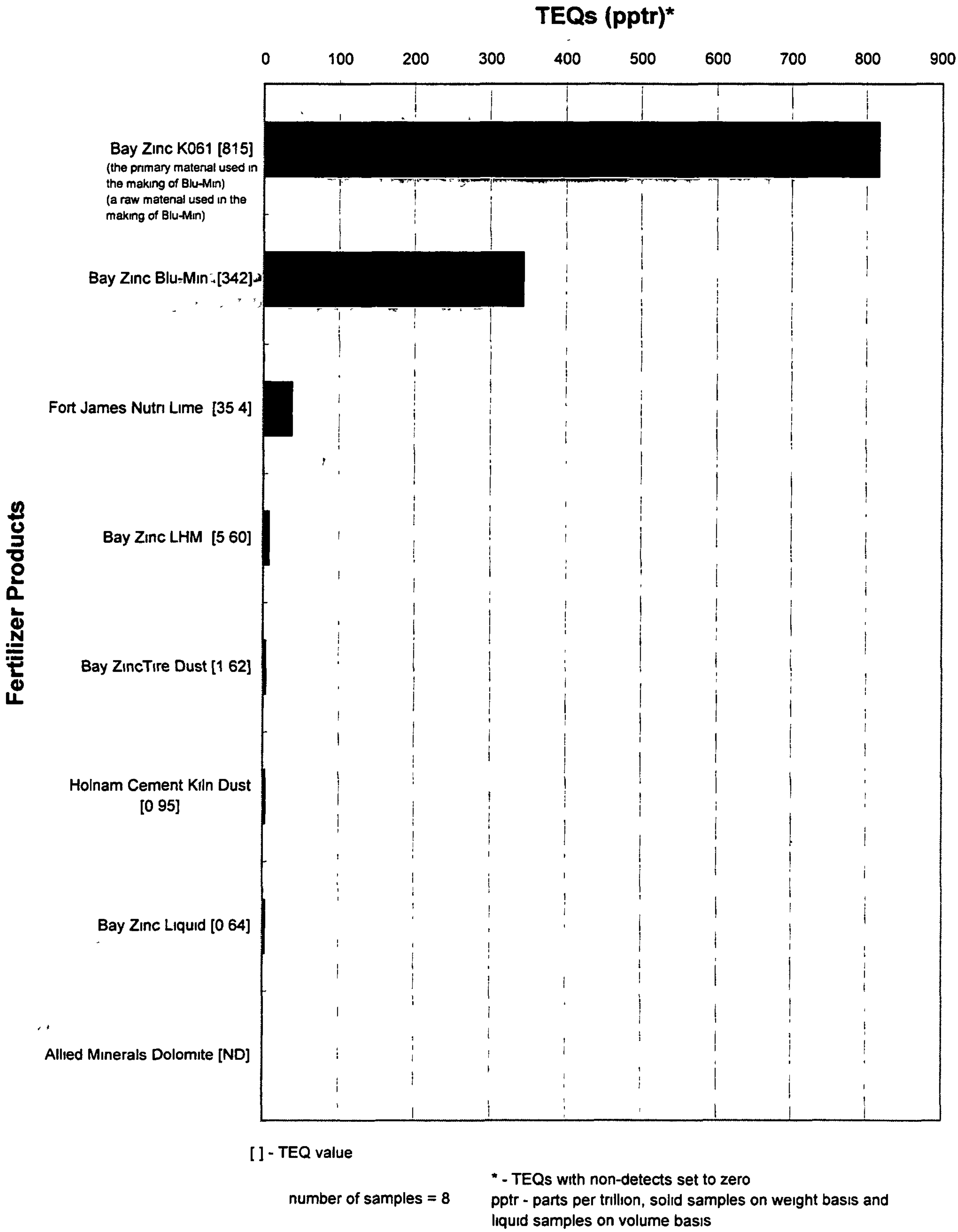

Figure 1-2. Rank-ordered dioxin TEQs in fertilizer products and fertilizer source materials - 1997 sampling results.

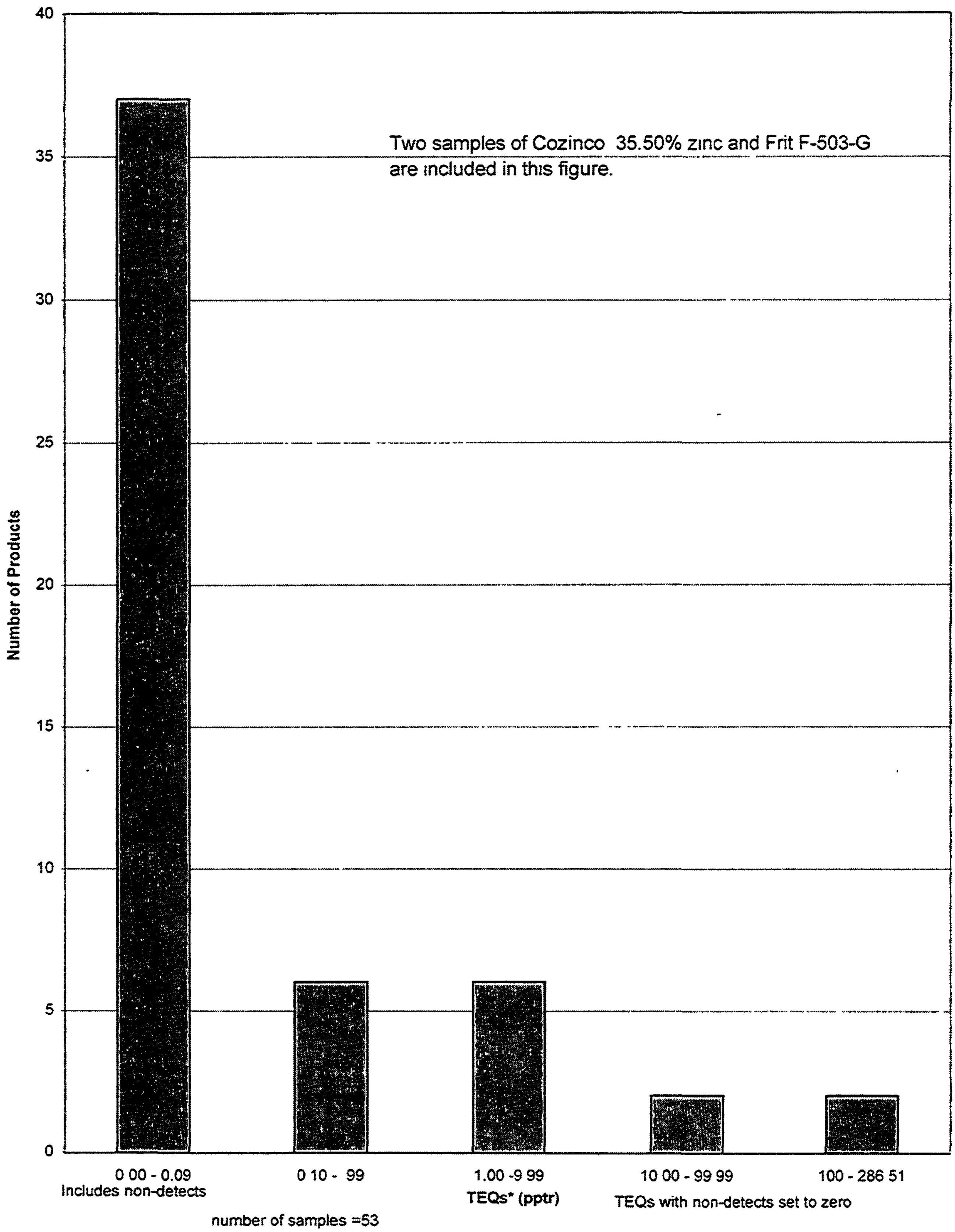

Figure 1-3. Frequency distribution: dioxin TEQs in fertilizer products - 1998 sampling results.

Conclusions

Some fertilizer products had higher concentrations of arsenic, cadmium, lead, mercury and selenium than others Currently Ecology is in the process of obtaining application rate information for these products to determine whether they fail the Washington Standards adopted in RCW 15 54 800.

Cadmium was found in relatively high concentrations in two phosphate fertilizers tested Cadmium was also elevated in agricultural soils as compared with background soils in the Metals in Soils Study (Chapter 2).

Seven of the 51 products tested in 1998 exceeded TCLP limits, all for cadmium These products were (1) Agrium Ammonium Phosphate Sulfate, (2) United Agri Products 0-45-0, (3) Frit F-503G, (4) Webfoot Rhododendron, Camelia, and Azalea Food, (5) NuLife 10-20-20, (6) NuLife Agro 10-15-10, and (7) Thrifty Payless Tomato and Vegetable Food.

Seventy-one percent of the 51 fertilizer products tested in the 1998 study had dioxin TEQs of less than 0 1 pptr Most of the fertilizers sampled contained less dioxin in its toxic form than did the soils surveyed (Chapter 3) Three of the fertilizer products sampled in 1998 and two of the fertilizer products sampled in 1997 had TEQs higher than any of the TEQs found in the soils dioxin study A literature review found no studies of dioxin levels in fertilizers There are no applicable standards for dioxin in fertilizers at this time

A few fertilizer products were found to contain relatively high levels of dioxin Of those sampled in 1998, fertilizer products having TEQs greater than 50 pptr were (1) Frit F-420G (287 pptr), (2) Frit F-503G (85 9 pptr mean value), and (3) NuLife All-Purpose Trace Elements (53 7 pptr) By comparison, the highest TEQ found of all waste-derived fertilizer products and micronutrients sampled in 1997 was Bay Zinc's 18% Blu-Min micronutrient, with a TEQ of 342 pptr The Fort James Nutri Lime sample in 1997 was found to have a TEQ of 35 4 pptr

2. Metals in Soils

Purpose

The objective of the metals in soils study is to determine if certain metals have accumulated in agricultural soils of the Columbia Basin of Washington State.

Ecology analyzed seven metal concentrations in agricultural and non-agricultural (background) soils from the Columbia Basin Irrigation Project and compared the results with three other soil studies Agricultural lands are defined as lands in active agricultural production Non-agricultural lands are lands that have never been farmed or tilled

This study, funded by *EPA Surveys, Studies, Investigations and Special Purpose Grant #X-980130-01-0,* will give the Legislature and others information to determine if further fertilizer regulation is needed

Sampling Procedures

The Columbia Basin Irrigation Project was selected for soil sampling because of the agricultural diversity and potential availability of historical information. The study area included the portions of Adams, Franklin, and Grant counties within the Columbia Basin Irrigation Project An important aspect of this study area is that historical agricultural practices (e g., cropping patterns and agricultural chemical use) can be documented in this area

Fields with historical use of biosolids (sewage sludge) and/or lead arsenate pesticides were excluded from this study, and sampling was limited to irrigated agricultural fields Background sites were non-irrigated areas Twenty agricultural sites and 13 matched background non-agricultural sites were sampled

Site History

Grant County is located in central Washington State and covers approximately 691,175 hectares (1,707,870 acres) The Columbia River flows in a deep valley along the southwestern boundary of the county The southern portion of the county contains Saddle Mountains and Frenchman Hills. Babcock Ridge and Beezly Hills border the northern part of the plain (Gentry, 1984)

Grant County has approximately 62 types of soil with a wide range of texture and natural drainage Soil blowing and water erosion are major soil-related problems in the southern part of the county Agriculture is the main economic enterprise in the county. About 19 percent of the total area is irrigated cropland, about 18 percent is non-irrigated cropland, and about 62 percent is rangeland Rangeland includes natural grasslands, savannas, wetlands, deserts, and areas that support certain forb and shrub communities (Gentry, 1984) Only 971 hectares (2400 acres or

0 1% of the county) are classified as urban The county's main irrigated crops are winter wheat, alfalfa hay, potatoes, corn, and beans (Gentry, 1984) The main non-irrigated crop is winter wheat

Site Selection

The Columbia Basin Irrigation Project is divided into uniquely numbered farm units. Each farm unit represents one or more fields owned by an individual Computer generated random numbers were used to create a list of potential farm unit numbers Potential farm unit numbers corresponding to actual farm units on farm unit maps were verified (USDI, 1982).

Landowners were contacted and asked to participate in the study Before owners were contacted, fields were roughly compared to selection criteria and U S. Department of Agriculture (USDA) soil survey maps (Gentry, 1984) Selection criteria for agricultural field sampling followed Holmgren site selection criteria (Holmgren et al , 1993) See Appendix 2-A for selection criteria

If a farm unit did not meet the selection criteria, it was eliminated from the list of potential sampling locations before contact with the owner When owners were contacted, selection criteria were confirmed Sites not meeting the criteria were excluded from the study before participation was requested If available, the historical agricultural use of acceptable sites was recorded See Figure 2-1 for map of generalized sampling locations

Participation in the sampling program was voluntary To judge whether voluntary participation might introduce a bias toward fields lower in heavy metals, the rejection rate was recorded (Appendix 2-B) As owners were asked to participate, their response was recorded Twenty rejections (prior to obtaining 20 participants) were the predetermined rejection rate that would constitute an unacceptable, unquantifiable bias Had 20 rejections been obtained, this study would have been terminated

At the time the twentieth landowner agreed to participate, eight had rejected the opportunity Landowners did not exhibit a bias related to knowledge about metal concentrations in the agricultural soil Several participants acknowledged some reservation about the sampling because the heavy metal content of their soil was unknown to them, although they routinely tested the soil for nutrient content

Matching Background Sites with Agricultural Fields

The agricultural sites selected all had potential background sites in close proximity However, in several cases, additional background sites were located because access could not be obtained for sampling Some background sites were farther from the agricultural sites than originally planned Appendix 2-C lists soil types and the distances separating the agricultural sites from the background sites In all cases soil types of the selected agricultural and background sites were identified using USDA soil maps

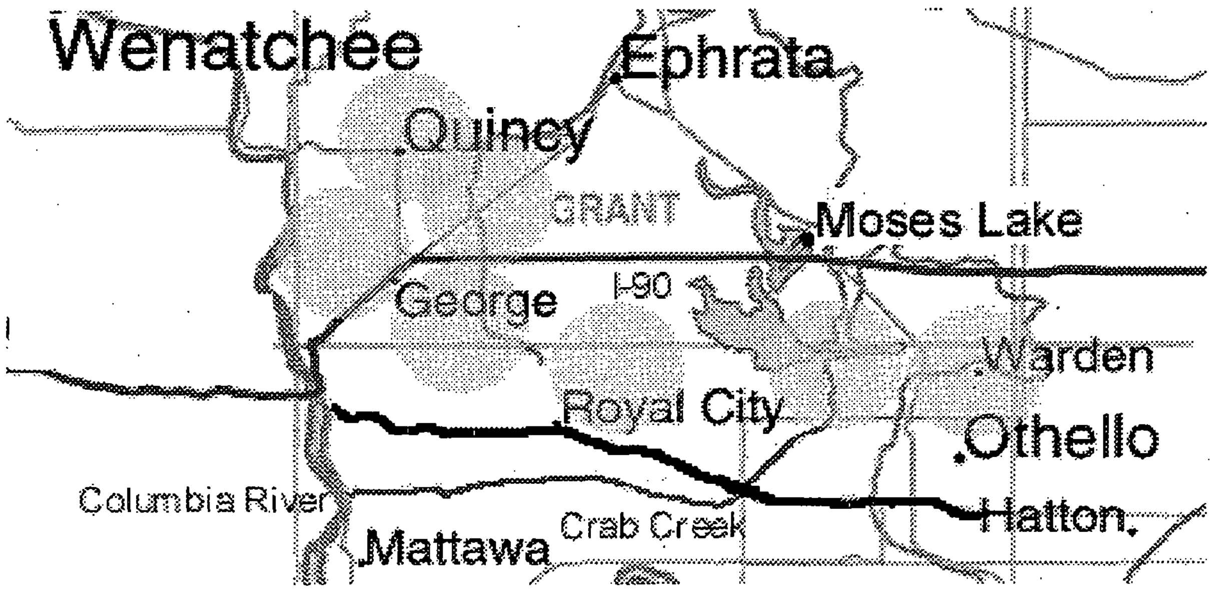

Figure 2-1. Map of general sampling locations.

The background sites were selected based on landowners' site history and visual evidence they had not been cultivated. Ten background sites had sagebrush (*Artemesia sp.*) and cryptobiotic crusts present. Two sites had 1.83 m (6 foot) tall sagebrush specimens. and steep topography that suggested no historical cultivation. One site was within an historic railroad right-of-way and had been plowed for weed control by the second-generation landowner but never planted. A more certain background site could not be found for this soil type due to its presence in prime farmland. See Appendix 2-D for soil particle size distribution per sample and soil type.

Table 2-1 summarizes the crop types associated with the sites sampled in this study. Compared with the USDA Soil Survey (Gentry, 1984), the crop types encountered are representative of the area. They also represent a variety of farming practices (e.g., row crops, small grains, orchard). Additionally, dry land or non-irrigated farming practices may have been used on some lands before irrigation began in the 1950s.

The U.S. Geological Survey (USGS) Soil Survey for Grant County (Gentry, 1984) was used to classify soil types for all sites. Table 2-2 summarizes the soil types by sample site. The table also lists the percent of that soil type found in Grant County. A brief description of each soil type can be found in Appendix 2-E.

Table 2-1. Crop types on fields sampled.

Crop Type on Fields Sampled	Number of Fields Sampled	Main Irrigated Crops (Gentry, 1984)
Alfalfa	7	Alfalfa
Apples	3	---
Beans	2	Beans
Corn	1	Corn
Oats	1	---
Pasture	1	---
Potato	1	Potato
Primrose	1	---
Sugar Beet	1	---
Wheat	2	Winter Wheat

Table 2-2. Soil types of fields sampled in the Columbia Basin.

Percent (%) of Grant County	Soil Type and Number of Samples	Percent (%) of Total Sample Sites
5%	Ephrata-Malaga	9%
	Ephrata fine sandy loam (3)	
11%	Kennewick-Warden-Sagemoor	40%
	Kennewick fine sandy loam (2)	
	Kennewick silt loam (4)	
	Warden silt loam (3)	
	Sagemoor silt loam (2)	
	Novark silt loam (2)	
12%	Quincy	12%
	Quincy fine sand (4)	
6%	Taunton-Scoon	6%
	Scoon silt loam (2)	
4%	Shano	15%
	Shano silt loam (3)	
	Prosser very fine sandy loam (2)	
4%	Timmerman-Quincy	18%
	Timmerman coarse sandy loam (4)	
	Royal very fine sandy loam (2)	

Sampling Procedures

For each site sampled, large-scale maps of the selected fields were obtained, and 0.4 hectare (one-acre) grid was used with a global positioning system (GPS) to identify the latitude and longitude of the starting point in each one-acre sampling unit.

Five samples were taken within the identified one-acre sampling unit and combined to create one composite sample per sampling unit or field The starting point was one sample, with the other four samples collected in a radius originating from the starting point at a distance of approximately 27 4 meters (30 yards) at equal intervals of 90°.

The surface layer of vegetative or organic material was removed and a 30 5-cm (12-inch) deep hole was dug. A depth of 30.5 cm was used to account for local tilling and farmers' nutrient-sampling practices Equal portions of soil were collected with a clean stainless steel trowel between the surface and a depth of 30 5 cm from an uncontaminated side of the hole An equal amount of material was removed from each sample site and combined in a clean stainless steel mixing bowl Samples were thoroughly mixed and placed in precleaned sample jars

Sample jars were labeled with an Ecology seal, a sample number, date and the investigator's initials. All samples were stored in a cooler and maintained at a temperature of 4°C until analyses Chain-of-custody procedures followed Manchester Environmental Laboratory (1994a) guidelines The samples for this project were delivered to the Manchester Laboratory by Ecology staff.

Field quality assurance consisted of four "blind" replicate samples taken from the same agricultural field Blind replicate samples are identical samples submitted to the laboratory with different identification numbers. An estimate of the combined sampling and laboratory precision can be determined by calculating the relative percent difference (RPD) between the duplicate sample results The RPD is the ratio of the difference and the mean of the results expressed as a percentage

Analyses

Appendix 2-F summarizes the analyses and methods used on each sample Analyses conducted on all samples included, pH, soil particle size (or grain analysis), total organic carbon (TOC), total available phosphorous, and cation exchange capacity (CEC) In addition to total metals analyses (As, Cd, Cu, Pb, Hg, Ni, Zn), an extraction procedure and analysis to help determine metal concentrations available to plants was also performed (DTPA extractable metals As, Cd, Cu, Pb, and Zn) The intent of the DTPA procedure was to determine the portion of metals present that may be available to plant life (Spielman and Shelton, 1989)

Data Quality

Precision and Accuracy

Routine laboratory quality control procedures were adequate to estimate laboratory precision and accuracy for this project Laboratory quality control tests were done on each set of 20 or fewer samples and consisted of blanks, duplicate samples, and spiked samples

One sample in each set of evaluations was analyzed in duplicate in order to assess precision Precision and accuracy for all analyses was also assessed through the analysis of two matrix spikes and matrix spike duplicates. Method criteria were applied to results to ensure acceptable accuracy and precision

Representativeness

Because a comprehensive analysis of area soils was not cost-effective at the time of this study, the results are considered a screening survey of the area and not a characterization of the Columbia Basin Irrigation Project soils. The sample size determination was arbitrary

Comparability

Samples were analyzed using standard analytical methods (US EPA SW-846 methods) at the Manchester Environmental Laboratory and state-accredited laboratories Samples were analyzed for metal concentrations at the 1 0 mg/kg detection level, or lower. Metals not detected or "non-detects" were identified as appropriate

General Chemistry Quality Assurance

The data generated by the analysis of these samples can be used with the qualifications discussed in Appendix 2-G.

Metals Analysis Quality Assurance

The data generated by the analysis of these samples can be used noting data qualifications discussed in Appendix 2-H.

Data Limitations

Field variability (variability within a single field) was not addressed in this study Only one composite sample per field was taken Limitations, particularly for site matching, in these analyses are due to differences in soil properties and only having limited resources to look at cursory soil properties Active agricultural fields and background sites also differ by irrigation practices The small sample size was also a limiting factor

Results and Discussion

Results

Data Summary

Table 2-3 summarizes the results of the metals in Columbia Basin Irrigation Project Agricultural soils Samples below the detection limit for a specific metal were not included in the arithmetic mean calculations. This had little effect on the total metals results DTPA extraction results had many non-detects; as a consequence, the means are artificially elevated Results include 20 agricultural samples and 13 background samples For the complete data set, see Appendix 2-I

Table 2-3. Summary statistics of Columbia Basin soil analyses.

Analysis	Agricultural Field Mean[1,4]	Agricultural Field Ranges	Background Mean[4,5]	Background Ranges
Total Metals (mg/Kg dw)				
Arsenic	3 35	2 10-5 68	2 91	1 50-5 56
Cadmium	0 103	0 050-0 210	0 060 (n=11)	0 030-0 098
Copper	14 3	9 49-19 0	13 5	9 89-20 2
Mercury	0.008 (n=18)	0 003-0 013	0 012 (n=12)	0 0032-0 066
Nickel	11 3	7 90-15 7	10.6	8.00-14 1
Lead	7 28	5 78-9 59	6 75	4.60-9.97
Zinc	53.1	43 6-65.0	45 6	32.5-56 2
DTPA Extraction (mg/Kg dw)				
Arsenic	0.577(n=7)	0 480-0 740	0 53(n=1)	0 480-0 530
Cadmium	0.077(n=16)	0 040-0 130	0 059(n=3)	0.040-0 080
Copper	2.84	1.43-4 85	2 48	0 072-4 71
Lead	0 797	0 230-1 52	0.676(n=12)	0 160-1 36
Zinc	3 91	0 320-6.97	1 34	0 0670-3 64
Total Organic Carbon (TOC)				
@ 104C (%)	0 660	0 330-1 14	0 530	0 210-1 06
@ 70C (%)	0 630	0.300-1 06	0 510	0 190-1 06
pH	6.94	5 50-7 95	7 51	6 40-8 20
Phosphorus (mg/Kg dw[2])	804	618-1060	846	587-1460
Cation Exchange Capacity (CEC) (g/Kg ww[3])	3 63	2 52-5 33	3 45	2 21-4 92
CEC mg/100 g soil	15 8	11 0-23 1	15 0	9 61-21 4

[1]N=20, unless otherwise specified
[2]Dry weight
[3]Wet Weight
[4] Non-detects were not included in arithmetic mean calculations
[5] N=13, unless otherwise specified

Statistical Summary

The sample collection design for analyzing metals in agricultural soils attempted to provide "paired" or matched samples from agricultural fields and non-farmed background fields. A paired sampling design is beneficial to the degree that it controls for extraneous variability that tends to mask the effects of a targeted variable The pairing of samples needs to control for the extraneous factors that could affect measurements, by making the members of each pair "equal" on most traits To detect an effect without controlling extraneous variability, much larger sample sizes may be required. A paired sampling design allows for a smaller sample size while maintaining statistical power to detect effects, or (equivalently) greater power can be achieved for a fixed number of samples

One method for pairing of soil samples would be to sample in the same fields before and after application of fertilizers or other soil amendments and growing crops That design could not be implemented for this screening study Instead, the pairing of soil samples in this study was based on matching of soil types and spatial proximity between paired background and agricultural fields While many of the background samples were adjacent to the agricultural field samples, several were located at a considerable distance relative to the overall study area dimensions (Appendix 2-C) It is also uncertain in some cases whether or not the agricultural field samples and matched background samples are identical in soil type These factors raise some concerns about the strength of the pairing in the study as carried out, even though the two data sets (agricultural fields and background fields) are far from independent. The study design as carried out probably represents an intermediate condition between independent and a paired sample design

Given the reasonable questions about the strength of pairing between samples, statistical analyses were first performed using an approach with minimal assumptions about the data sets All statistical analyses were conducted using SYSTAT 7 01 (SPSS, 1997). Nonparametric, unpaired statistical tests comparing agricultural field results and background field results were first performed using the Mann-Whitney (Wilcoxon) rank sum test This approach assumes the two data sets are independent and does no require that data be normally distributed The non-parametric test is based on the ranks of the measurements in the combined data sets The null hypotheses that there is no difference between agricultural and background fields was tested, against the alternative hypothesis that there is a difference (two-tailed test) The results are provided in Table 2-4 Statistically significant differences (p less than 0 05) are shown for cadmium and zinc, as extractable (DTPA) cadmium and zinc Geometric means for the data sets are listed in Table 2-5, non-detects were included in this calculation as a value equal to the detection level Cadmium and zinc total metals and DTPA were higher in agricultural soils than in background soils

A second set of statistical analysis was performed assuming that the data are paired, using the two-tailed, nonparametric Wilcoxon signed rank paired test For these tests, whenever more than one agricultural field was paired with a background field, the data from the multiple matched agricultural fields were averaged This produced 13 pairs of matched results A statistically significant difference was found between agricultural and background fields for cadmium and zinc, as well as DTPA zinc (results now shown here)

Table 2-4. Statistical summary of data.

Analysis	Mann-Whitney U Test Statistic	Probability
Total Metals (mg/Kg dw[1]) Arsenic	114 0	0 555
Cadmium	30 5	**<0.000262**[4]
Copper	100 0	0.269
Mercury	119 5	0 699
Nickel	106 5	0 386
Lead	100 5	0.277
Zinc	60.0	**0.010**[4]
DTPA Extraction (mg/Kg dw) Arsenic	93 0	0 07
Cadmium	43 0	**0.001**[4]
Copper	96 5	0 217
Lead	94 0	0 185
Zinc	30 0	**<0.0002459**[4]
Total Organic Carbon (TOC) @ 104C (%)	104 5	0.347
@ 70C (%)	109 0	0 439
pH	170 0	0.14
Phosphorus (mg/Kg dw)	119.5	0 699
Cation Exchange Capacity (CEC) (g/Kg ww[2])	118.0	0 658

[1]Dry weight
[2]Wet weight
[4]Bolded numbers indicate a statistically significant difference

Table 2-5. Geometric means of metal concentrations in soils.[1]

Analysis	Agricultural Field Mean[2]	Background Mean[3]
Total Metals (mg/Kg dw[4]) Arsenic	3.21	2.76
Cadmium	0.096	0 059
Copper	14.1	13 3
Mercury	0.008	0.01
Nickel	11.1	10 4
Lead	7.20	6 60
Zinc	52.8	45 0
DTPA Extraction (mg/Kg dw) Arsenic	0 572	0 53
Cadmium	0 074	0 064
Copper	2 74	1 90
Lead	0 729	0 611
Zinc	3 37	0 925
Total Organic Carbon (TOC) @ 104C (%)	0 621	0 477
@ 70C (%)	0 587	0 460
pH	6 90	7 49
Phosphorus (mg/Kg dw)	793	809
Cation Exchange Capacity (CEC) (g/Kg ww[5])	3 57	3 40

[1]Non-detects were not included in geometric mean calculations
[2]Number of samples = 20
[3]Number of samples = 13
[4]Dry weight
[5]Wet weight

Discussion

Only cadmium and zinc concentrations are significantly higher in agricultural samples than background samples, however, arsenic, copper, nickel, and lead mean concentrations appear higher in agricultural samples than background samples (Table 2-3, Table 2-5). The data indicate agricultural practices have impacted soils over the past 50 years Soils concentrations of all metals in this study were typically less than or at the lower ranges of the comparison studies data See figures in Appendix 2-J for a graphical comparison of these data to the studies discussed below

In order to put these results in context, the data were compared to three studies

- Natural Background Soil Metal concentrations in Washington State (Ecology, 1994a),
- Background Concentrations of Metals in Soils from Selected Regions in the State of Washington (Ames & Prych, 1995), and
- Cadmium, Lead, Zinc, Copper, and Nickel in Agricultural Soils of the United States of America (Holmgren et al , 1993)

It is important to understand the differences in these studies in order to evaluate the results Numerical comparisons of these studies are found in Table 2-6

The Ecology (1994) study determined "natural background" concentrations of metals in Washington State soils. Samples were collected statewide, by region The two most comparable regions to this study were·

- Yakima Basin (Yakima, Kittitas, Chelan, and Grant counties), and
- Group "E" (Benton, Spokane, Lincoln, Adams, Okanogan, and Whitman counties)

Soil samples were collected from predominant soil series and efforts were made to collect samples from undisturbed or undeveloped areas (Ecology, 1994) Samples were collected from the "A," "B," and "C" soil horizons or from ground surface to a depth of about 91 cm (3 feet) (Ecology, 1994a) This is a distinct sampling difference with this study, as Ecology (1994) sampled about 61 cm (24 inches) deeper (Horizon A) and in different soil horizons (Horizons A-C) The same analytical methods were used in both studies

The Washington Department of Ecology and the US Geological Survey investigated the magnitude and variability of background metal concentrations in state soils (Ames & Prych, 1995) Sampling procedures in Ames & Prych (1995) were similar to those used in this study (Horizon A) with one significant difference, samples were collected between 61 and 96 5 cm (Horizons B and C) Soil series sampled were Quincy, Shano, and Taunton Region R (central Columbia Basin) in the Ames & Prych (1995) study was used for comparison to this study The same methods and analyses were used in the Ames and Prych study as in this study

The Holmgren et al (1993) study analyzed 3,045 surface soil samples from 307 different soil types for several metals, including cadmium and zinc, throughout the United States. The primary purpose of their study was to assess the background levels of cadmium and lead in major food crops and in the soils of their major growing areas Sample depth was 50 cm as opposed to the 30 5 cm sample depth used in our study Ecology obtained county level data from the primary author (Chaney, 1998) The arithmetic and geometric means for Grant and Adams county data are presented in Table 2-6 and graphically compared with the other studies in Appendix 2-J

Table 2-6. Comparison of arithmetic and geometric means of metal concentrations in soils.

Soil Studies	Arithmetic Mean (mg/Kg)						
	As	**Cd**	**Cu**	**Pb**	**Hg**	**Ni**	**Zn**
Agricultural Soils	3 35	0.103	14 3	7.28	0 008	11.3	53.1
Background Soils	2 91	0 06	13.5	6 75	0 012	10 6	45 6
Ecology (1994b) Yakima Basin	3 73	0.55	20.16	7.03	0 03	24 83	57 54
Ecology (1994b) Group "E"	2.7	<0 20*	17.69	6 92	0.01	13 77	45.74
Ames & Prych (1995)	3.4	<0.20*	20	7	0.027	25	50
Holmgren et al (1993)*	NA	0.170	22 7	7.44	NA	20.5	63 5
	Geometric Mean (mg/Kg)						
Agricultural Soils	3 21	0 096	14 1	7 2	0 008	11 1	52 8
Background Soils	2 76	0 059	13 3	6 60	0 01	10 4	45
Holmgren (1993) (n=122)	NA	0 184	26 7	8 5	NA	26 4	66
Ames & Prych (1995) (n=60)	<2 8	<0 20	17	7	<0 016	17	47

*Grant and Adams Counties only

Cadmium levels were less than 0 21 mg/Kg in all agricultural sites and less than 0 098 mg/Kg in all background sites This corresponds with the Ecology (1994) study in which all Group "E" cadmium values were below detection limits (0.2 mg/kg) Ames & Prych (1995) also found all cadmium concentrations below detection limits (0 2 mg/Kg) Cadmium values in the Holmgren data from Grant and Adams counties ranged from 0.1 to 0 26 mg/Kg and were closer to the results of this study These studies imply that cadmium soil concentrations in the Columbia Basin are typically below 0.26 mg/Kg

Zinc concentrations in this study ranged from 43 6 to 65 0 mg/Kg in agricultural fields, with an arithmetic mean of 53.1 mg/Kg The Ecology (1994b) study reported similar values for Group E and the Yakima Basin Ames & Prych (1995) found zinc concentrations from 21.0 to 116 mg/Kg with an arithmetic mean of 50.0 mg/Kg Zinc concentrations in Grant and Adams county (Holmgren 1998) were also very similar to this study See Table 2-6 for a comparison of means between studies and Appendix 2-J for graphical representations of data ranges in these studies

Metal concentrations for arsenic, copper, lead, mercury, and nickel from our study were also very comparable with the above studies (see Table 2-6 and Appendix 2-J), with similar values and within the same ranges

Crop uptake potential for cadmium (a contaminant often found in phosphate fertilizers) is a concern because of the potential problems of cadmium in plants used as food sources Although cadmium is ubiquitous in the environment, uncontaminated baseline levels in soil pose little risk to human and ecosystem health (Gavi et al, 1997, Chaney et al 1996)

Certain pairs of metals, such as zinc and cadmium, compete with one another for plant absorption (Felsot, 1997) The crustal ratio of zinc to cadmium is often determined and used when discussing plant uptake of these metals. Zinc is a plant nutrient and necessary for normal plant growth (Amrani et al , 1997) Generally, if enough zinc is present (e.g., when the zinc-to-cadmium ratio is high), plants will preferentially absorb zinc before they absorb cadmium With adequate zinc present in the agricultural and non-agricultural soils, the potential for excess cadmium in crops is very low

The data for our study shows that zinc-to-cadmium ratios are relatively high, just as Holmgren et al (1993) found That study showed that the zinc-to-cadmium ratios in Washington were some of the highest in the country, Holmgren considered ratios over 350 to be very high (Holmgren et al , 1993)

Zinc-to-cadmium ratios were calculated for agricultural fields and ranged from 252 to 1160 The average zinc to cadmium ratio for the agricultural fields was 592 These ratios were also calculated for background sites and ranged from 460 to 1780 with an average of 979

As noted above, our data indicate that over the last 50 years agricultural practices have increased cadmium concentrations over background levels in the Columbia basin While we do not know the quality of the fertilizers used in the past, of the fertilizers sampled by this study, the highest cadmium levels were in the phosphate fertilizers. Cadmium is a known contaminant of phosphate fertilizers (Holmgren, et al, 1993, Chaney & Oliver, 1994).

Although the standards adopted in the past year by Washington State limit the amount of cadmium in fertilizers, these standards still allow for some increase of metals in the soil over time It is known the current rate of increase or how many years are required before these metals, particularly cadmium, will approach levels of concern

To study the eco-toxicity and mobility of metals in soils and assess the available metal fraction to plant life, single-extraction tests, such as diethylenetriaminepentaacetic acid (DTPA), are commonly used (Quevauviller *et al*, 1998) According to a micronutrient rating schedule used to evaluate soils for adequate micronutrients available to plants (Waddoups, 1996), the DTPA extraction values (Table 2-3) for zinc in agricultural samples indicate very high levels of zinc available to plants

Additionally, the DTPA-zinc values for agricultural fields are statistically significant from the background DTPA-zinc levels DTPA results also indicate that zinc appears highly available to plants in both the agricultural sites (mean of 3.91 mg/Kg or ppm) and the background sites (mean of 1 34 mg/Kg or ppm) Zinc values of 1 0 to 2 0 ppm are considered moderate to high values. A zinc value of 3 00 or greater is considered very high too excessive or toxic (Waddoups, 1996)

DTPA-copper results indicate that a high amount of copper is potentially available for plant uptake Waddoups (1996) considered copper values of 3.0+ mg/Kg or ppm to be toxic or excessive and 0 4 to 0 6 mg/Kg or ppm to be a moderate to high level Not enough information is known about cadmium DTPA to make any determinations Many other factors, such as pH, are associated with the plant uptake of micronutrients

The ability of a soil to absorb and desorb micronutrient elements is the cation exchange capacity (CEC) The unit of chemical measurement used is milliequivalents (meq) per 100 grams soil (Waddoups, 1996) CEC values generally range from 2 to 10 meq for sandy textured soils, from 12 to 20 meq for silt loams, and above 20 meq for heavy clay and highly organic soils The exchange capacity is nearly a fixed value for a specific soil, changing only with time intervals (e g., 20-50 years) or as a result of a major change such as heavy erosion or deposition (Waddoups, 1996).

In the original study design, this measurement was to be used to help identify soil series (Table 2-2) This information gives additional evidence that soil series were correctly, if approximately, identified In this study, CECs (Table 2-3) averaged 3 63 g/Kg ww or 15 8 meq/100g soil at agricultural soils and 3 45 g/Kg ww or 15 0 meq/100g soil for background soils No statistical differences were found between agricultural and background sites Since the soils tested were primarily silt loams, both values are within the range (12-20 meq/100g soil) for the soil types sampled

Conclusions

Cadmium and zinc concentrations are significantly higher in agricultural fields than in the background sites, presumably due to farming practices used over the last 50 years These increased cadmium and zinc concentrations do not indicate any potential increased risk to the environment, since the values detected are within the lower range of background comparison studies (Ecology,1994; Holmgren, *et al,* 1993, and Ames & Prych, 1995) Results from zinc-to-cadmium ratios indicate zinc may be more available to plants than cadmium in both the agricultural and non-agricultural sites sampled Adverse environmental and human health effects due to cadmium uptake in plants would not be expected

Increased agricultural cadmium levels over background indicate a need to monitor these metals over a period of time to determine their rate of increase and ensure levels do not become a concern While the Washington State fertilizer standards, adopted earlier this year, limit cadmium levels in fertilizers, they still allow for increases of metals in the soil Chapter 1 describes several fertilizers, several of which are potentially waste-derived, with high levels of cadmium These include United Agricultural Products (UAP) 0-45-0, Frit F-503G, Agrium Ammonium Phosphate Sulfate, NuLife Agro 10-15-10, and three home and garden products No other soil metal concentrations were detected that could potentially be attributable to the metal concentrations found in fertilizers

The levels of arsenic, copper, mercury, nickel, and lead in soils do not show significant differences between agricultural fields and matched background sites The mean values of arsenic, lead, and zinc from agricultural fields and background sites are very similar to Ecology (1994), Ames & Prych (1995), and Holmgren *et al* (1993) studies on metal concentrations in Washington state soils Copper, mercury, and nickel values for this study are lower than the results from the other studies (Ecology,1994b, Holmgren *et al,* 1993; and Ames & Prych, 1995)

3. Dioxins in Soils

Purpose

The objective of this study is to provide an initial assessment of typical dioxin concentrations in soils in Washington State, particularly agricultural soils

Ecology sampled soils in open, forested, and urban areas to determine if dioxins occur in these areas and at what levels The original study design included sampling and analyzing agricultural soils for dioxins However, due to difficulties in randomly selecting agricultural sampling sites and an inability to guarantee confidentiality to landowners, agricultural soil sampling and analysis was not conducted this year

Study Design

Open Area Soils

Eight samples were collected from "open" areas For this study, open areas were defined as historically non-forested, non-agricultural, and located away from large urban areas Because a total of only eight sites were located in open areas, no attempt was made to choose the samples randomly Sites were chosen based on spatial distribution (four samples each, from east and west sides of the state) and ability to gain site access Four of these samples were collected from grazed land, and four from conservation areas and reserves

Two sites in eastern Washington were sampled to represent grazed land Both sites were on state lands managed by the state Department of Natural Resources One site was located near Palouse Falls, and the other sample was collected from rangeland northeast of Ellensburg In western Washington a sample was collected from a horse ranch in Clark County, and the other sample was collected from a dairy farm in Pierce County

Two sites on each side of the state were sampled to represent open, non-grazed land Three of these sites were from national wildlife refuges, and the fourth sample was collected from a national park

Forest Soils

Eight soil samples were collected from "forested" areas Forested sites were defined as areas that have an extensive canopy composed primarily of mature trees Because a total of only eight sites were located in forested areas, no attempt was made to choose the samples randomly. Sites were chosen based on spatial distribution (e g , east and west sides of the state) and ability to gain site access

Four samples were collected from areas actively managed for silviculture (e.g , sites that have been logged and are slated for future logging) Soil samples were obtained from both private and public forests Two samples were collected from public forests, Wenatchee National Forest and Olympic National Forest The other two samples were from private forests, one near the town of Newport and one near the town of Rainier.

Four soil samples were obtained from forested areas that had not been managed for timber harvest Collection sites were (1) a state park in the southeast corner of the state, (2) Olympic National Park, (3) Willapa National Wildlife Refuge, and (4) Pasayten Wilderness in the Okanogan National Forest.

Urban Soils

Fourteen of the 30 soil samples collected were allocated to urban areas As defined by the U S Census Bureau (1997), an urbanized area comprises one or more places ("central place") and the adjacent densely settled surrounding territories ("urban fringe") that together have a minimum of 50,000 persons The urban fringe generally consists of contiguous territory having a density of at least 1,000 persons per 256 hectares (one square mile) According to the U S Census Bureau (1998) Washington has 10 urbanized areas, comprising a total of 3,394 square kilometers

To allocate 14 sites within the urban areas, a random number generator was used on a database of 3,394 units based on one-kilometer square grids representing the total urban area Random numbers were used to allocate the sample count among the urban areas, sequential numbers were used to represent the size, but not specific locations, within the listed urban areas of the state The database was then sorted by these random numbers. The first 14 units in the list determined the urban areas selected for this study Since each square kilometer was not assigned specifically to an exact location other than by urban area name, samples sites were listed by urban name only (Appendix 3-A)

The Seattle urban area has the greatest land area and population The majority (9) of the urban samples were collected within this area. Using maps that defined urbanized areas, Ecology randomly selected public sites such as "parks" and other similar grass-covered landscapes for this study "Parks" were used because they are generally not in industrial areas or close to point sources of dioxins, they tend to be in residential areas and at least one-acre (0 4-hectare) Approximately 300 sites listed as parks in the Seattle urban area were entered into a database (Thomas Brothers Maps, 1989) To select these nine sites within the Seattle urban area, a random number generator function was used on the database Each park was assigned a random number and the database was then sorted by these random numbers The first nine parks (excluding those in the city of Seattle) in the list that met the selection criteria were the sites selected for soil sampling. Seattle parks were not sampled due to (1) difficulty in getting timely information and (2) a lengthy permit process

Due to the small sample sizes allocated to the other urban areas (1-2 soil samples), no attempt was made to randomize site selection in these areas Sites selected were in residential areas within the urban boundaries

Agricultural Soils

Ecology encountered significant problems obtaining samples from agricultural lands, and found it impossible to gain permission to sample randomly selected agricultural sites within study time constraints Ecology could not guarantee sampling results would be confidential, and property owners were reluctant to have their soils tested As a result, sampling of agricultural land was postponed Ecology is exploring ways to sample agricultural land and assure that (1) samples are random and (2) locations at which samples are collected are kept confidential

This study is limited to the 30 soil samples allocated to open, forested, and urban areas

Sampling Procedures

A global positioning system (GPS) was used to identify the starting point and sampling locations on the selected property An attempt was made to avoid locating sampling sites near roads, railroad tracks, treated wood utility poles or fences, or areas of significant erosion

A sampling unit of one-acre (0 4-hectare) was selected, because this was the largest practical unit that allowed for representative composite sampling For example, finding an urban area of ten acres (4 05 hectares) suitable for sampling proved difficult A one-acre site allowed for uniform sized sampling units across all land uses

Samples were collected based, in part, upon guidelines developed for the EPA National Dioxin Study (EPA 1984) and other published studies Each sample was a composite of ten samples collected within the sampling unit The initial sample was collected at a starting point, with nine additional samples collected at the end of a radius originating from the starting point, extending a distance of 36 m (39 yards) and rotated at equal intervals of 40° The surface layer of organic vegetative material was removed, and a sample was collected from a depth of 0-5 cm below the surface to include an equal amount of material throughout the depth of the sample. Dioxins are relatively immobile substances and do not appreciably leach through the soil Each sample contained approximately 120 cm^3 (6 ounces) of material, and was collected using dedicated utensils Sampling apparatus was appropriately cleaned prior to sampling (Appendix 1-C)

Samples were collected with a stainless steel scoop, placed in a stainless steel mixing bowl, and thoroughly mixed Composite soil samples were mixed until the entire sample was uniformly consistent Rocks, vegetation, and debris were removed from the samples in the field using stainless steel tweezers Samples were placed in ultra-clean sample jars with a Teflon lid for transport and analyses Each sample was analyzed for dioxin, total organic carbon (TOC), and grain size All samples were stored in a cooler and maintained at a temperature of 4° C until analysis A summary of sample handling procedures for dioxin samples is found in Table 3-1

Table 3-1. Dioxin analyses, container, and holding summary.

Target Analyses	Minimum Sample Size	Holding Requirements
2,3,7,8-substituted PCDD/PCDF Method 8290 (EPA 1994)	Min. 10g sample in ultra clean glass jars with Teflon lids	Cool to 4°C and keep dark; max. hold 30 days[2]; analyze within 45 days of extraction

Sample labeling, shipping, and chain-of-custody procedures as defined in the Manchester Environmental Laboratory Lab Users Manual (Ecology, 1994) were followed

Analytical Procedures

Analysis of 2,3,7,8-substituted PCDD/PCDF congeners (forms) was conducted at MAXIM Technologies Inc /Pace Analytical, using high resolution GC/MS EPA Method 8290, with enhancements derived from Method 1613B

Detection limits varied depending on the physical state (e g , moisture content, organic content) of the samples, but the target detection limit was 0 1 pptr EPA Method 8290, Section 7 9 5, specifies the sample specific Estimated Detection Limit (EDL) as the concentration of a given analyte required to produce a signal with a peak height of at least 2 5 times the background signal level Not all the congeners were responsive enough to provide EDLs at 0 1 pptr

Data Quality

This project is designed to provide data on "typical" concentrations of dioxins in Washington State soils. It is primarily for informative and descriptive purposes, and does not test any specific hypotheses The study was not designed to test whether specific land uses are associated with significant differences in dioxin levels, but general comparisons can be made among land uses

Representativeness

This study was designed to generate representative data Spatial stratification (e g , samples taken from locations across the state and across several land uses) provides adequate coverage and data consistent with pilot study objectives Representativeness was enhanced through use of composite sampling However, conclusions based on data generated by this pilot study are limited given the non-random sampling of open and forestlands, as well as small sample sizes

[2] The holding time of 30 days from collection to extraction is a recommendation. PCDDs and PCDFs are very stable in a variety of matrices, and holding times for samples stored at 4°C in the dark may be as high as a year for certain matrices.

Quality Control Procedures

Established laboratory quality control procedures met data quality objectives for laboratory precision and accuracy for this project Laboratory quality control tests were done on each set of 20 or fewer samples and consisted of blanks, duplicate samples, and spiked samples Manchester Laboratory quality control samples and procedures are discussed in Manchester Environmental Laboratory Lab Users Manual (Ecology, 1994)

Quality assurance and quality control measures indicate dioxin results are reliable A number of the 17 congeners were detected in the associated method blank at concentrations below the lowest calibration standard According to the method, re-analysis is not required when a target congener is detected below the lowest calibration standard If the concentration of a congener in a sample was less than five times the method blank, a qualifier was added to the result specifying that the analyte was not detected at or above the (estimated) reported result. In cases where the sample concentration for a congener was greater than five times that of the method blank, the blank result is considered insignificant relative to the concentrations detected in the samples

Field quality assurance for this project consisted of two duplicate split samples The differences in duplicate sample results reflect combined sampling and laboratory variability The Richland split samples were relatively close, with TEQs of 4 50 and 4.75 pptr. The split sample from Spokane had TEQs of 0 33 and 0.98 pptr.

Sample 98328339 was re-analyzed because only one of the 15 internal standards met the recovery criteria

For further details on quality control procedures see Appendix 3-B

Results and Discussion

All data within the text, tables, and figures are based on TEQ values with non-detects assumed to equal zero (ND = 0) TEQ refers to the total equivalent toxicity of dioxin congeners (the different forms of dioxins and furans present) calculated as shown in Appendix 3-C (Yake et al , 1998) See Appendix 1 for a discussion on TEQs and how they are calculated

A summary of the dioxin data is listed in Table 3-2 TEQs for each sample are listed in Appendix 3-D Complete analytical results for the 17 congeners of concern for dioxins and furans can be found in Appendix 3-D, TOC and grain-size results are located in Appendix 3-E Figure 3-1 shows TEQs of the dioxin soil analyses by land use Every sample had detectable levels of dioxins, even samples from remote wilderness areas Dioxins are ubiquitous, they are found throughout the state, most likely as a result of aerial deposition (Czuczwa et al , 1984, Czuczwa and Hites, 1986, Creaser et al , 1989, Rotard et al , 1994)

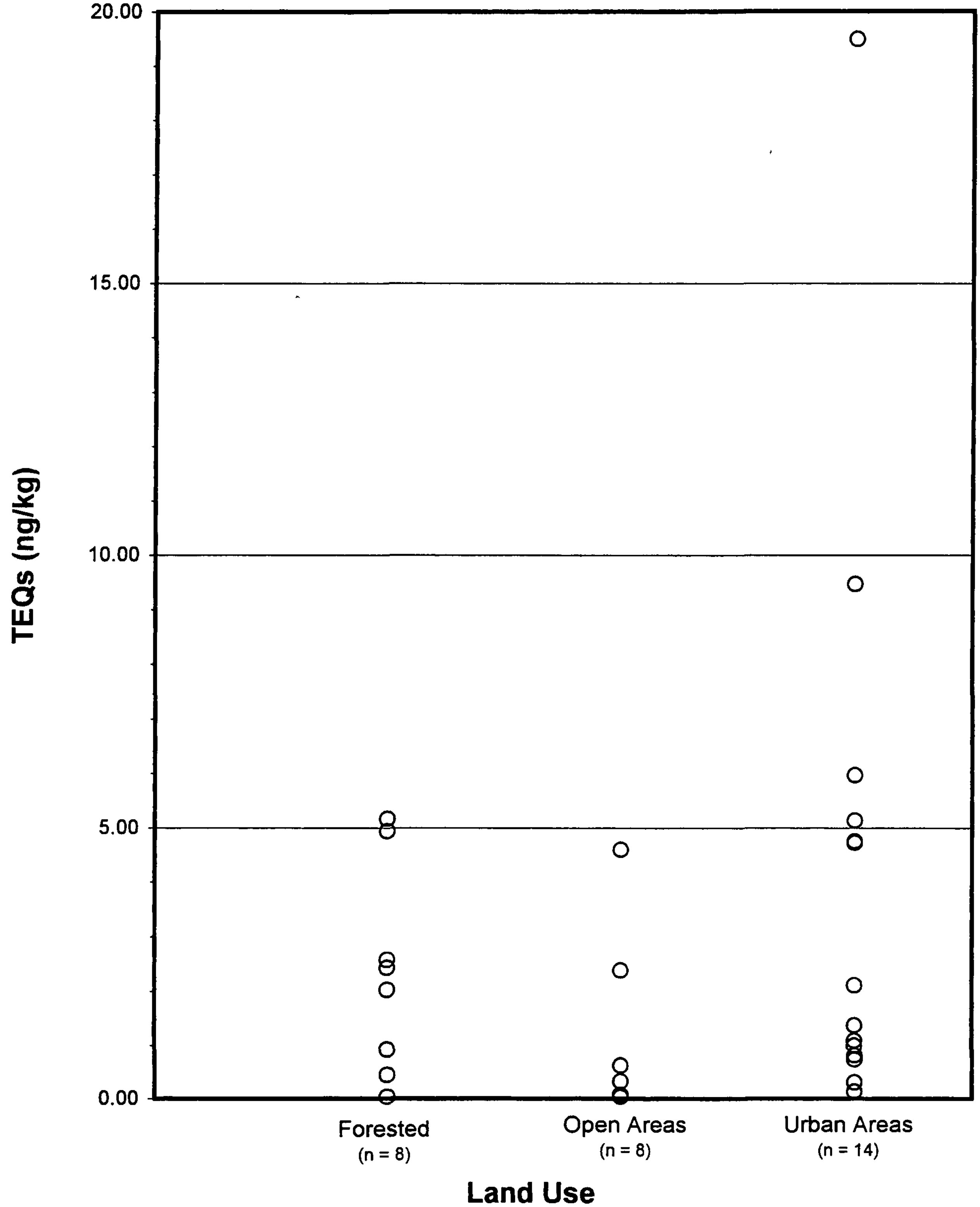

Figure 3-1. Dioxin TEQs of select soils in Washington State by land use.

Table 3-2. Summary of Washington State soil TEQs* by land use. (pptr)

Land Use		Range	Mean	Median	Geometric Mean	N
Urban		0. 133 – 19.5	4.07	1.73	1.92	14
	Tri-Cities	1 44 - 4 75	3 10	3 10		2
	Spokane	0.984	--	--		1
	Tacoma	9 47 - 19 5	14 5	14 5		2
	Seattle	0 133 - 5 96	2 36	1 36		9
Open		0.0400 – 4.59	1.01	0.27	0 248	8
	Grazed	0.0400 – 4.59	1.32	0 33		4
	Non-grazed	0 0460 – 2.37	0.71	0 21		4
Forest		0.033 – 5 16	2.31	2.22	1.20	8
	Commercial	0.033 – 2.42	1.35	1 47		4
	Non-commercial	0.45 – 5.16	3.28	3 75		4
Total		0 033 – 19.5	2 79	1.22	0.983	30

*Toxicity Equivalent

Most of the data is centered around the low end of the scale with a few higher levels This type of data distribution results in an inflated mean that is not truly representative of the central tendency of the data The median is the middle value, with an equal number of points above and below this value The geometric mean is often used to describe the central point of data that has this type of distribution If the data were normally distributed, all of these measures of central tendency would be approximately equal

The range of results from urban areas is greater than that of the other two land use areas Urban areas also include the three sites with the highest soil TEQs These results are consistent with other studies comparing TEQs of urban lands to rural lands in Austria (Boos et al., 1992), Britain (Creaser et al , 1990) and Spain (Schuhmacher et al , 1997)

There is no accepted background standard for dioxin levels with which to compare these data Little support can be found for attributing dioxins to natural processes As a result, this study addresses typical rather than a natural “background” level Very few published studies are available on typical levels of dioxin in United States soils for comparison Most published studies on dioxin in soils in the U.S are associated with potential sources (e.g , incinerators) With many of the published studies, it is very difficult to accurately determine how the TEQs were calculated, making direct comparisons difficult However, a few similar studies conducted in Europe can be used as a rough comparison The TEQ soil levels found in this study are within the range of similar studies conducted in other countries (Figure 3-2)

The data plotted for Spain comes from two studies (Jiménez et al., 1996, Schuhmacher et al , 1997) For a more direct comparison, data from urban and rural areas in Spain are plotted by dry weight, and not normalized to TOC Dioxin concentrations are often correlated to organic content In this study the correlation coefficient between dioxin (TEQ) and TOC of 0.34 (log – log correlation) approached but did not meet statistical significance at the 0.05 level ($p = 0.066$). Also, in Spain TEQ values near an incinerator were calculated using half the value of the detection limit for congeners not detected (Jiménez et al., 1996). The median values from Germany (Rotard et al., 1994) are based on international TEQs, the report does not state whether congeners below the detection limit were given a value of zero for TEQ calculations. The sites sampled in Germany were outside of industrial and urban areas.

Dioxins are unintended industrial byproducts from processes such as combustion (Czuczwa & Hites, 1986, Creaser et al , 1990; Alcock & Jones, 1996), therefore, the sites closest to these sources (e g , incinerators, industrial boilers, and cement kilns) have some of the highest dioxin concentrations A comparison between the east and west side of the state (Table 3-3), excluding urban samples, shows that the sites sampled in eastern Washington tend to have lower levels of dioxin in the soils (Figure 3-3) This effect may be attributed to more people and greater development (i e., urban areas) in western Washington

Table 3-3. Summary of soil dioxin data (expressed as TEQs) by east and west side of the state, excluding urban samples. (pptr)

Land Use	Range	Median	Mean	Geometric Mean	N
East	0.0330 – 5.16	0 0645	0 846	0 161	8
West	0 330 – 4.93	2 40	2.48	1.85	8

The two soil samples collected from Tacoma have the highest levels of dioxin detected (19 5 and 9 47 pptr TEQ) in this study Although the Tacoma sites are located in residential areas, they may be located closer to industrial areas than the other urban sites sampled Possible historical sources of dioxin in Tacoma include hog fuel boilers, smelters, pulp and paper mills, as well as municipal and other incinerators Tacoma has several cleanup sites with confirmed dioxin contamination (Yake et al , 1998)

Forest sites appear to have dioxin levels greater than open areas In Germany forests have some of the highest levels of dioxins (Rotard et al , 1994) (Figure 3-2) Trees and vegetation may act as a large filter (Rotard et al , 1994, Horstmann et al , 1997) resulting in a greater surface area available for dioxin absorption and deposition than an open grassy area The leaves and needles accumulate gaseous and particulate dioxins from the atmosphere, resulting in greater deposition of dioxins on the forest floor (Horstmann et al , 1997) In addition, organic matter in a forest is not harvested or removed as frequently as it is in areas managed for timber or agriculture. This may account for the apparent difference between commercial forests and the wilderness areas, although a small sample size precludes making a definitive statement

Average TEQ values appear to be higher for grazed lands than non-grazed lands (Table 3-2) The median values of these areas are too close, and there are too few samples to speculate about potential differences in dioxin levels of grazed and non-grazed lands

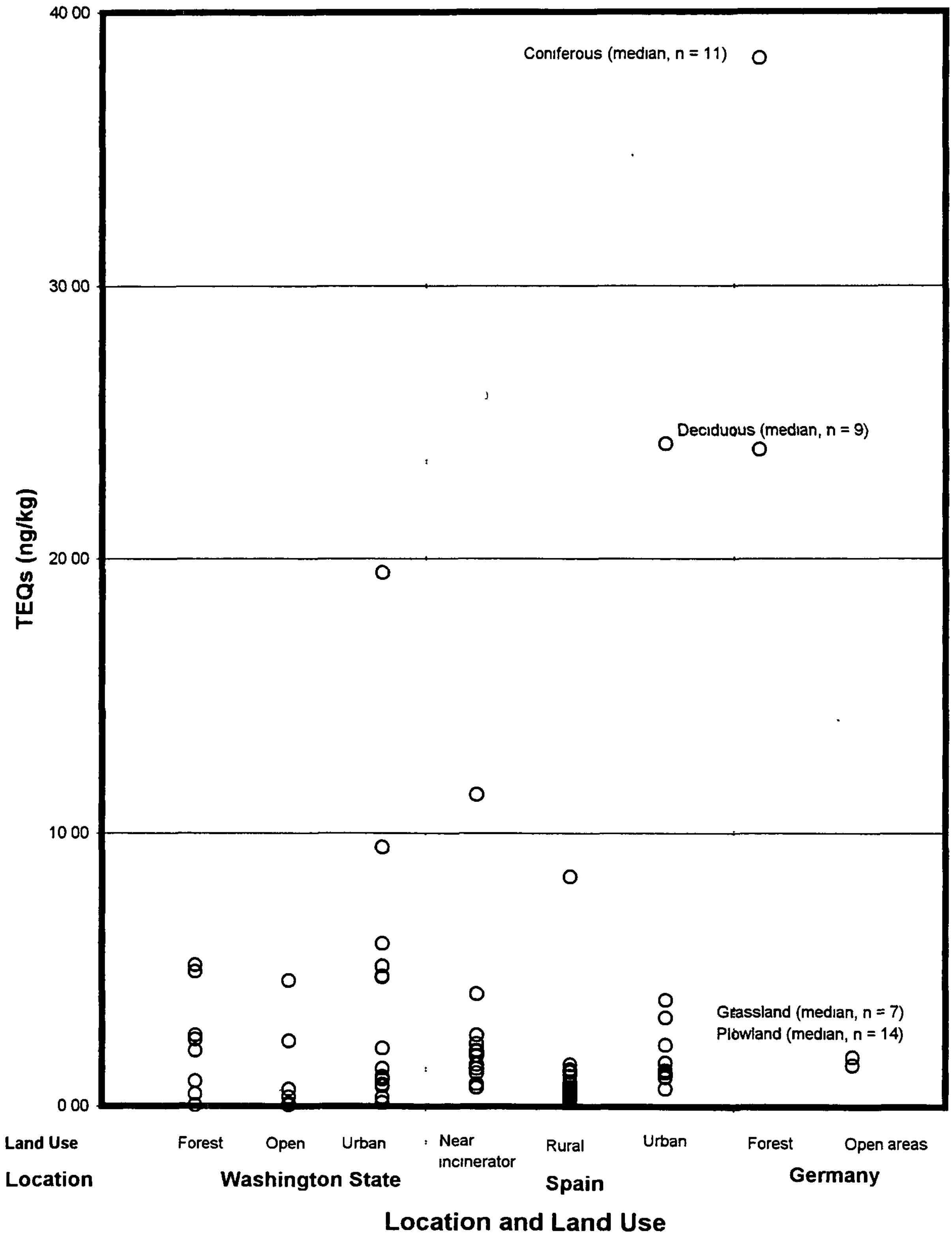

Figure 3-2. Dioxin TEQs of soil by select land uses in Washington State, Spain and Germany

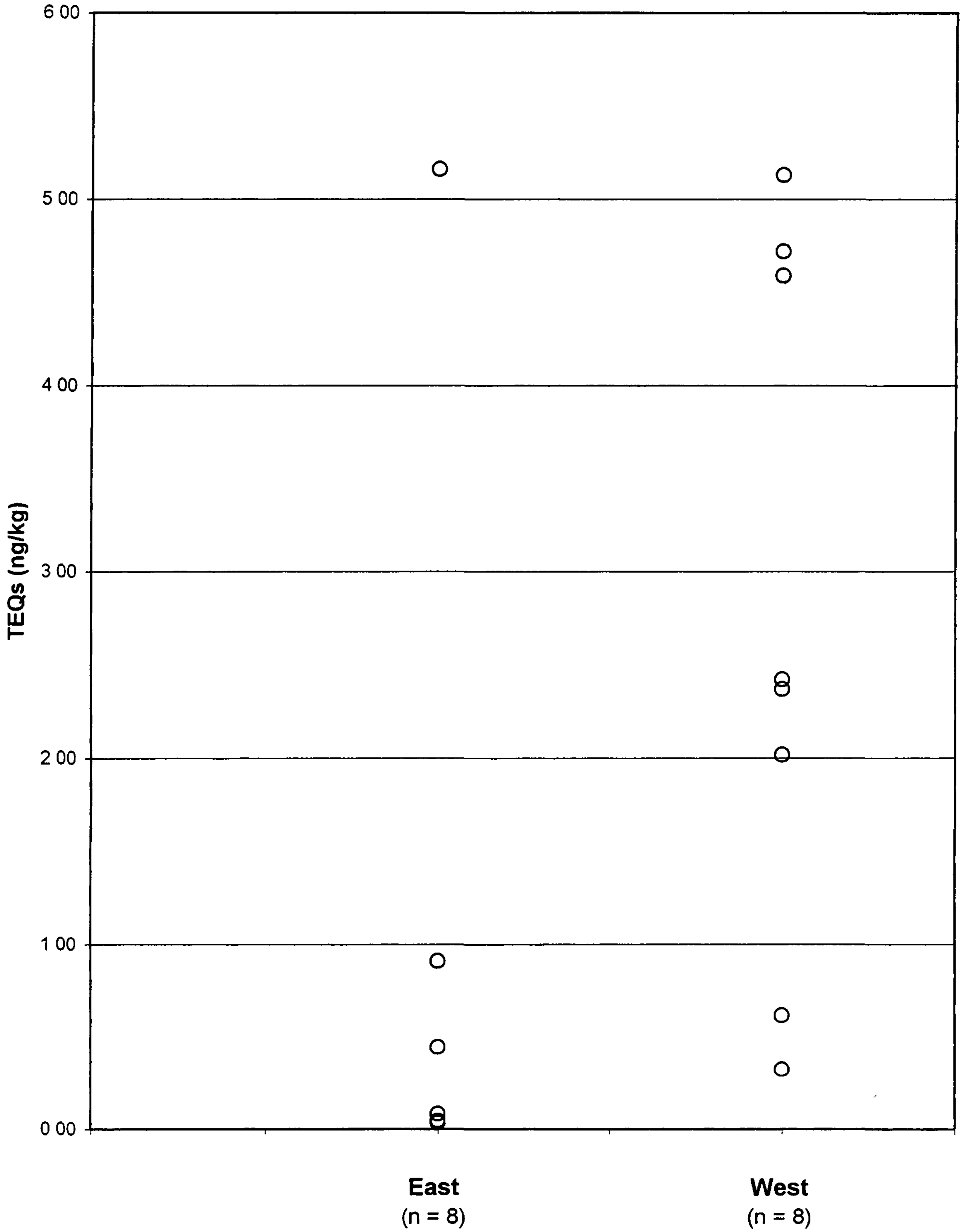

Figure 3-3. Dioxin TEQs of soil samples in Eastern and Western Washington State excluding urban areas.

Conclusions

Ecology was unable to conduct the study as originally designed This study will not be completed until Ecology collects and analyzes soil samples from agricultural lands for dioxin, scheduled for 1999 This study was not designed to statistically compare dioxin levels among different land uses, but generalizations can be made about dioxin levels in Washington State

Dioxins are found in surface soils throughout the state They were detected in all samples, including samples from remote wilderness areas supposedly far removed from human influences The overall range of dioxin TEQs in soils sampled were from 0.033 to 19 5 pptr, the median value was 1 22 pptr

The three highest values of dioxins were from urban areas. Urban areas also had the greatest range of TEQ values (0 133 - 19.5 pptr) Forested areas appear to have a higher level of dioxins in soils than open areas; however, more samples are required to provide a more definitive answer

The results of this study indicate that the levels of dioxins detected in Washington State are comparable to other parts of the world

Conclusions, Policy Options, and Recommendations

Conclusions

This preliminary report includes a description and findings for three studies (1) metals and dioxins in fertilizer products, (2) metals in soils, and (3) dioxins in soils These studies provide a better understanding of contaminants in fertilizer products and how some fertilizer components may be affecting Washington State soils.

1. Metals and Dioxins in Fertilizer Products

The objective of this study is to quantify metals and dioxins in fertilizer products Ecology randomly sampled and analyzed 51 bulk agricultural and home-use fertilizers, as well as micronutrients and a soil amendment, to determine their dioxin concentrations The samples were also analyzed to determine heavy metal concentrations

Metals in fertilizer products

Of the 51 fertilizer products tested, seven failed Ecology's new screening criteria for waste-derived fertilizer Two bulk or packaged agricultural fertilizers, one agricultural micronutrient fertilizer, and four home-use packaged fertilizers failed the toxicity characteristic leaching procedure (TCLP) for cadmium Concentrations of cadmium in the TCLP test ranged from 1 5 to 2.52 ppm, compared to a criterion of 1 0 ppm

Fertilizers likely to have been derived from steel mill flue dust (K061) were among those with the highest levels of metals K061 is exempt from hazardous waste requirements if used to make zinc-containing fertilizer. Five of the seven fertilizers with high levels of cadmium (relative to the others tested) are potentially waste-derived, and three of the five are likely to be exempt. Ecology is concerned about fertilizers that fail the dangerous waste tests, because it may indicate that these fertilizers are not in compliance with Washington State regulations

Some fertilizer products had higher concentrations of arsenic, cadmium, lead, mercury, and selenium than others Currently, Ecology is in the process of obtaining application rate information for these products to determine whether they fail the 1998 Washington Standards for the maximum allowable addition of metals to soil

Dioxins in fertilizer products

Most of the 51 fertilizer products tested contained non-detectable or extremely low levels of dioxin, more than 70% had dioxin toxic equivalents (TEQs) of less than one-tenth of one part per trillion (pptr) A few bulk agricultural fertilizers contained relatively high levels of dioxin Two fertilizers had TEQs greater than 140 pptr, and one product exceeded 50 pptr. Most conventional fertilizer products, as opposed to waste-derived fertilizers, had virtually no detectable levels of dioxins The three products with comparatively high levels of dioxin all appear to be derived from steel mill flue dust, K061

2. Metals in Soils

The objective of this study is to determine if certain metals have accumulated in agricultural soils of the Columbia Basin Ecology analyzed agricultural and non-agricultural (background) soils from the Columbia Basin Irrigation Project for seven metal concentrations and compared the results with two other state soil studies Thirty-three sites were sampled in the Columbia Basin Twenty samples came from agricultural soils and 13 from non-agricultural soils

The data indicate that agricultural practices in Washington State over a period of 50 years may result in increased concentrations of some metals in agricultural soils A statistical difference was found in the amount of zinc (a nutrient) and cadmium (a contaminant) in agricultural soils versus the amount found in non-agricultural soils Both zinc and cadmium levels are higher in the agricultural soils. These increased cadmium and zinc levels do not indicate any increased risks for human health and the environment The ratio of zinc to cadmium, which favors zinc uptake before cadmium, indicates that cadmium levels do not pose a problem in plant uptake Zinc is an essential element required for normal plant and animal life All metal concentrations, including the highest values found for zinc and cadmium in the agricultural soil samples, are at the low end of the range for Washington State soils

No significant difference was detected in the levels of arsenic, mercury, nickel, copper, and lead between agricultural soils and non-agricultural soils The results of this study (specifically the relationship between metals in fertilizers and metals in soils) were consistent with what Ecology expected to find, based on information obtained in 1997 (Bowhay et al , 1997)

3. Dioxins in Soils

The objective of this study is to provide an initial assessment of typical dioxin concentrations in soils in Washington State The original study design included sampling and analyzing agricultural soils for dioxins However, due to difficulties in randomly selecting agricultural sampling sites and an inability to guarantee confidentiality to landowners, agricultural soil sampling and analysis was not conducted this year Ecology plans to complete the agricultural soil sampling in 1999

Ecology obtained 30 soil samples in open, forested, and urban areas to determine if dioxins occur in these areas and at what levels Dioxins are found throughout Washington State Dioxin values ranged from 0 033 to 19 5 TEQ (ng/kg) All samples had detectable levels of dioxin, including samples from remote wilderness areas In general, average dioxin levels appear to be higher in urban areas than forested and open areas Three of the highest values detected were in urban areas This was expected since the primary source of dioxins is from combustion processes The results of this study indicate that the levels of dioxins detected in Washington State are comparable to other parts of the world

Policy Options for Regulating Bioaccumulative Chemicals of Concern in Fertilizers

Ecology has proposed the goal of virtually eliminating the release of all toxic, persistent, and bioaccumulative chemicals of concern (BCC) in the environment by the year 2025 It is known that at least three BCCs are found in some fertilizer products dioxins, cadmium, and mercury. While Washington recently adopted standards for cadmium and mercury in fertilizer, the state does not have standards for dioxins In addition, no standards exist for dioxins in fertilizer products in other states or countries

This discussion focuses on regulating dioxins in fertilizer, however, most of these policy options would be applicable for cadmium and mercury as well Many of the options may be interim steps toward a goal of BCC-free fertilizers by 2025. Ecology options are

- Require fertilizer companies to test and report levels of dioxins to Ecology without mandating a standard (to obtain more information about current levels before phasing in a standard)
- Reward companies with publicity/awards for manufacturing and selling dioxin-free or BCC-free fertilizers
- Encourage EPA to address this issue nationally
- Eliminate the steel mill flue dust (K061) exemption in the state Dangerous Waste Regulations (WAC 173-303)
- Evaluate elimination of the wood ash exemption in the state Dangerous Waste Regulations (WAC 173-303)
- Amend the Dangerous Waste Regulations and set a dioxin standard for waste-derived fertilizers Possible standards are
  - ◊ Non-detectable level of dioxins
  - ◊ "Background" or "typical" levels based on existing levels of dioxin in soil
  - ◊ Levels based on reasonable available technology to remove dioxins from fertilizers and their component sources
  - ◊ Levels that would eliminate the top 10% of highest dioxin concentrations found
  - ◊ Use of the EPA standard from cement kiln dust report (when final)
- Set a standard for dioxins in all hazardous waste
- Ask the Legislature to (1) set strict standards for BCCs (dioxins, cadmium, mercury) in all fertilizers or (2) ban all hazardous waste from being made into fertilizer

The above list applies primarily to fertilizers BCCs may also need to be addressed in soil amendments and other fertilizer products There are probably higher releases of BCCs from sources other than fertilizers and related products. Consequently, it may make more sense to address and eliminate the largest releases of BCCs into the environment before addressing BCCs in fertilizers However, the major route of BCC uptake by people is through food This may make the fertilizer issue a priority. Ecology will need to address these issues as the agency develops its implementation strategy for BCC elimination.

Recommendations

The following actions are recommendations for Ecology, as follow-up to this report.

- Continue to determine the levels of metals and BCCs (dioxins, cadmium, mercury) in fertilizer products, including levels of dioxins in agricultural soils, and share that information with the public in a timely manner
  - ◊ Work cooperatively with the state Department of Agriculture to implement the *Fertilizer Regulation Act*, by on-going review and sampling of fertilizer products for metals
  - ◊ Sample agricultural soils for dioxins during the spring of 1999
  - ◊ With input from stakeholders, determine (1) if more sampling of dioxins in fertilizers is needed, and (2) the need to obtain funding
- Review existing information about dioxins in biosolids and EPA's approach to this issue A proposed rule is due from EPA in December 1999, and will provide Ecology with additional direction in this manner
- Continue to encourage EPA to
  - ◊ Complete and release the dioxin risk assessment report and the cement kiln dust report
  - ◊ Adopt standards for hazardous waste-derived fertilizers and cement kiln dust used as fertilizers or soil amendments
  - ◊ Conduct an assessment of all fertilizer products, as well as related research, and develop risk-based standards
- Commit to a regulatory process to eliminate the steel mill flue dust (K061) exemption in Washington State
- Re-evaluate the wood ash exemption in the Washington State Dangerous Waste Regulations
- Work with stakeholders to develop a strategy to minimize BCCs and other metals of concern in fertilizer products
- With the state Department of Agriculture, in consultation with Department of Health and the Department of Labor and Industries, report progress in the biennial report to the Legislature on these recommendations about levels of non-nutrient substances in fertilizers (The first report is due December 1, 1999)

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Appendices

Appendix
for
Introduction

Appendix 1. Conventions used in calculating TEQs.

Dioxins occur in many forms or congeners Of the 210 congeners of polychlorinated dibenzo-*p*-dioxins and polychlorinated dibenzofurans (PCDDs/PCDFs), only 17 with chlorine atoms in the 2,3,7,8 positions are considered highly toxic (Birnbaum 1994) Seven PCDDs and ten PCDFs have this configuration. 2,3,7,8-tetrachlorodibenzo-*p*-dioxin (2,3,7,8-TCDD) is the most toxic While PCDDs and PCDFs with chlorine atoms in the 2,3,7,8 positions are considered the most toxic, 2,3,7,8-tetrachlorodibenzofuran (2,3,7,8-TCDF) is considered 1/10 as toxic as 2,3,7,8-TCDD The ratios adopted by international convention (EPA, 1989) are the basis for the calculation of TEQs as shown in the table below and in Appendices 1-N, 1-O, and 3-B TEQ is an overall measure of the toxicity of the forms present

Table 1. Toxicity equivalent factors (TEF) for the 17 PCDDs and PCDFs (EPA 1989).

	Congener	TEF
1	2,3,7,8-TCDD	1
2	1,2,3,7,8-PCDD	0.5
3	1,2,3,4,7,8-HxCDD	0 1
4	1,2,3,6,7,8-HxCDD	0 1
5	1,2,3,7,8,9-HxCDD	0.1
6	1,2,3,4,6,7,8-HpCDD	0 01
7	OCDD	0 001
8	2,3,7,8-TCDF	0 1
9	1,2,3,7,8-PCDF	0.05
10	2,3,4,7,8-PCDF	0 5
11	1,2,3,4,7,8-HxCDF	0.1
12	1,2,3,6,7,8-HxCDF	0.1
13	2,3,4,6,7,8-HxCDF	0.1
14	1,2,3,7,8,9-HxCDF	0.1
15	1,2,3,4,6,7,8-HpCDF	0.01
16	1,2,3,4,7,8,9-HpCDF	0.01
17	OCDF	0.001

EPA 1989 Interim procedures for estimating risks associated with exposures to mixtures of chlorinated dibenzo-p-dioxins and dibenzofurans (PCDDs and PCDFs) and 1989 update Prepared by the Risk Assessment Forum, Washington, DC EPA/625/3-89/016

When one or more of the 2,3,7,8-substituted dioxins or furans is not detected in the sample, the calculation of the toxicity equivalent (TEQ) for that sample can be ambiguous Three approaches to this dilemma are often used.

A One approach assumes that if a congener is not detected its concentration is zero The TEQ is calculated assuming no contribution from the undetected congeners (ND = 0) This approach yields the *minimum* value for the calculated TEQ

B A second approach assumes the congener(s) may have been present at concentrations as high as the detection limit. The TEQ is calculated assuming undetected congeners are present at the detection limit (ND = DL). This approach yields the *maximum* value for the calculated TEQ.

C A third approach takes the intermediate path and assumes undetected congeners are present at half the detection limit (ND = ½ DL) This approach yields a value for the calculated TEQ intermediate between the first and second method

The tables in the report text are based on TEQ values calculated using method A described above (e g , concentrations of undetected congeners are assumed to equal zero) Full analytical results and TEQ calculations for soil and fertilizer samples using these three methods are listed in Appendices 1-N, 1-O, and 3-B

The difference between minimum and maximum results for a single sample (methods A and B) ranged from 0 to 11 pptr for fertilizer samples and 0 to 8 pptr for soils The largest differences are generally found where the detection (or quantification) limits are relatively high High detection limits are often associated with samples high in complex organic matter from which it is difficult to extract dioxins The fertilizer samples with the highest differential between minimum and maximum calculated TEQ were Ponderay Newsprint Fiberay SC (consisting of paper fibers and wood chips) and Kelly Green Recycled Fresh Fish Both of these materials would be expected to have high concentrations of complex organic matter

Appendices

for

1. Metals and Dioxins in Fertilizers

Appendix 1- A. Soil amendments, fertilizers, and micronutrients - products sampled, 1998.

Soil Amendments

Manufacturer	Product Name	Grade/Constituents
Ponderay Newsprint	Fiberay SC	N/A

Bulk/Packaged Agricultural Fertilizers

Manufacturer	Product Name	Grade/Constituents
Northwest Alloys	High-Mag Gro Powder	1-0-8 +
		18% Magnesium*
		*(Magnesium analysis not guaranteed)
Fort James	NutriLime	12% equiv. CaCO3
		(OSU Method)
Unocal	Ammonium Nitrate	34-0-0
Agrium	Ammonium Phosphate Sulfate	16-20-0 +
		14% Sulfur
IMC Kalium	Potash	0-0-62.4
Global Recycling and Research	Kelly Green Recycled Fresh Fish	3-2-1
United Agri Products	UAP 0-45-0	0-45-0
High Yield Chemical Company	Sulfur	90% Sulfur

Agricultural Products with Micronutrients

Manufacturer	Product Name	Grade/Constituents
Frit	F-503G	2 40% Boron
	Sample #1	2 40% Copper
	Sample #2	14 40% Iron
		6.00% Manganese
		0.06% Molybdenum
		5.60% Zinc
Frit	F-420G	20% Zinc
RSA	Ruffin-Ready Zn	10% Zinc
Nutrient Technologies	Tech-Flo Zeta Zinc 22	22% Zinc
Stoller	Green Label Micronutrient II Super-Starter	1% Magnesium
		2% Copper
		1% Manganese
		4% Zinc
Cozinco	Zinc Sulfate Monohydrate	35 50% Zinc
	Sample #1 (Lab Log# 318085)	
	Sample #2 (Lab Log# 338201)	

Appendix 1-A - (cont'd) - Products Sampled, 1998.

Agricultural Products with Micronutrients (cont'd)

Manufacturer	Product Name	Grade/Constituents
Bioplus	Micro 700 Chelated Micronutrients	1-0-3 +
		1 00% Sulfur
		0 025% Boron
		0.07% Copper
		0.10% Iron
		0 70% Manganese
		0 0007% Molybdenum
		0.20% Zinc
Horizon Ag	Micro-Plus	2 00% Iron
		1.00% Manganese
		3 00% Zinc
Western Farm/Monteray	9% EDTA Zinc	9 00% Zinc
Monteray	Premium Zinc 10%	10 00% Zinc
Hydro-Agri/Viking Ship	FS/31 Ferrous Sulfate	31 00% Iron

Home-Use Packaged Fertilizer Products

Manufacturer	Product Name	Grade/Constituents
J R Simplot -	Best 6-20-20XB Premium Plant Food	6-20-20 +
Best Professional Products Division		1.50% Iron
		5 50% Sulfur
		0.75% Zinc
I.F M	Gaia's Own Cottonseed Meal	6-2-1
A.H Hoffman	Ace Tomato & Vegetable Food	8-10-8 +
		8% Calcium
		4% Magnesium
		3% Sulfur
		1% Iron
		0 2% Manganese
Northwest Chemical Corporation dba	Fred Meyer Moss Control-	12-2-4 +
United Horticultural Supply	Plus Lawn Food	18% Sulfur
		10% Iron
Northwest Chemical Corporation dba	Winter Green 15-10-25	15-10-25 +
United Horticultural Supply		3 60% Sulfur
		2% Iron

Appendix 1-A - (cont'd) - Products Sampled, 1998.

Home-Use Packaged Fertilizer Products (cont'd)

Manufacturer	Product Name	Grade/Constituents
Northwest Chemical Corporation dba	Webfoot Turf Treat 15-5-10	15-5-10 +
United Horticultural Supply		2.60% Sulfur
		0.0225% Boron
		3% Iron
		0.05% Manganese
		0.0006% Molybdenum
		0.055% Zinc
Terosa	Rose Food	5-8-2 +
		7.40% Calcium
		2.10% Magnesium
		3.70% Sulfur
		0.03% Boron
		0.40% Iron
		0.12% Manganese
		0.0012% Molybdenum
		0.10% Zinc
Evergro Products	Evergro 23-3-23	23-3-23 +
		2.10% Iron
Pursell Industries	Sta-Green Azalea, Camelia and Rhododendron Food	14-7-7 +
		0.02% Boron
		0.05% Copper
		1.00% Iron
		0.05% Manganese
		0.005% Molybdenum
		0.05% Zinc
Schultz	Schultz Bloom Plus	10-60-10 +
		0.10% Iron
		0.05% Manganese
		0.05% Zinc

Appendix 1-A - (cont'd) - Products Sampled, 1998.

Home-Use Packaged Fertilizer Products (cont'd)

Manufacturer	Product Name	Grade/Constituents
Northwest Chemical Corporation dba United Horticultural Supply	TurfGo	12-0-0 + 5.00% Sulfur 0.50% Magnesium 6 00% Iron 2 00% Manganese
Pace International	NuLife 10-20-20	10-20-20
Liquinox	Fully Chelated Iron and Zinc	0 20% Iron 0.20% Zinc
Pace International	McLendon Weed and Feed 15-5-5	15-5-5
Pace International	NuLIfe Agro 10-15-10	10-15-10 + 0.08% Zinc 4 05% Magnesium 10 00% Sulfur 0 03% Boron 0 03% Copper 0 21% Iron 0 09% Manganese 0.0009% Molybdenum
Pursell Industries	Sta-Green Nursery Special	12-6-6 + 0 02% Boron 0 05% Copper 0 25% Iron 0 05% Manganese 0 0005% Molybdenum 0.05% Zinc
QC	Ferrous Sulfate Monohydrate	30% Iron
Ampro Industries/Ringer	AmTurf Wildflower Mix/ Ringer Magic Start	1-1-1
Schultz	Schultz Soluble for Orchids	19-31-17 + 0 02% Boron 0 07% Copper 0 33% Iron 0 05% Manganese 0 0005% Molybdenum 0 07% Zinc
Scotts-Sierra Horticultural Products	Osmocote Vegetable and Bedding Plant Food	14-14-14

Appendix 1-A - (cont'd) - Products Sampled, 1998.

Home-Use Packaged Fertilizer Products (cont'd)

Manufacturer	Product Name	Grade/Constituents
Spectrum Group Division of United Industries Corp	Peter's Professional All-Purpose Plant Food	20-20-2 + 0.50% Magnesium 0 02% Boron 0 05% Copper 0 10% Iron 0 05% Manganese 0 0005% Molybdenum 0 05% Zinc
Chas H Lilly Co.	Thrifty Pay-Less Tomato and Vegetable Food	5-10-10
The Garden Grow Company	Whitney Farms 100% Organic Citrus, Berry & Vine Food	7-4-2
Solaris Group of the Monsanto Company	Ortho Upstart Vitamin B-1 Plant Starter with 3-10-3 Fertilizer	3-10-3
Pace International	NuLife All-Purpose Trace Elements	2 40% Boron 2 40% Copper 14 40% Iron 6.00% Manganese 0 06% Molybdenum 5 60% Zinc
The Garden Grow Company	Whitney Farms Jersey Green Sand	0-0-3
The Garden Grow Company	Whitney Farms Iron Sulfate	11% Sulfur 31% Iron
J R Simplot Co	Hydro-Feed with Polyon 20-10-10 Specialty Plant Food	20-10-10 6 00% Sulfur
Voluntary Purchasing Groups, Inc	Hi-Yield Pecan and Fruit Tree Fertilizer	12-4-4 + 1 00% Sulfur 1 00% Zinc
Northwest Chemical Corporation dba United Horticultural Supply	Webfoot Rhododendron, Camelia, Azalea Food	7-15-10 + 2% Calcium 1% Magnesium 4% Sulfur 0.0225% Boron 0 05% Copper 0 10% Iron 0 050% Manganese 0 0005 Molybdenum 0 060% Zinc
Ringer	Ringer Magic Start Grass Patch	0 5-1-1

Appendix 1-B. Cleaning procedures for metals and PCDD/PCDF sampling.

Sampling equipment cleaning procedures

1. Wash with laboratory detergent
2. Rinse several times with tap water
3. Rinse with 10% nitric acid solution
4. Rinse three times with distilled/deionized water
5. Rinse with high purity acetone
6. Rinse with high purity hexane
7. Allow to dry, and seal with aluminum foil

Appendix 1-C. Methods bibliography.

All methods are taken from Test Methods for Evaluating Solid Waste, SW-846, EPA, Office of Solid Waste and Emergency Response. Particular methods are as follows.

Laboratory Analysis	Method Used for Analysis
Acid Digestion of Sediments, Sludges, and Soils	Method 3050A. 7/92
Inductively Coupled Plasma-Atomic Emission Spectroscopy	Method 6010A. 7/92
Arsenic Atomic Absorption Furnace Technique	Method 7060A. 9/94
Selenium Atomic Absorption Furnace Technique	Method 7740. 9/86
Silver. Atomic Absorption Furnace Technique	Method 7761 7/92
Cadmium. Atomic Absorption Furnace Technique	Method 7131A. 9/94
Lead Atomic Absorption Furnace Technique	Method 7421 9/86
Mercury in solid or semi-solid waste (Cold Vapor Technique)	Method 7471A. 9/94
Toxicity Characteristic Leaching Procedure (TCLP)	Method 1311. 7/92
PCDD's and PCDF's by Hi-Res Mass Spectrometry	Method 8290. 9/94

Appendix 1-D. Quality assurance memos.

Appendix 1-D is included in a supplemental report, Ecology Publication 98-332:

<u>Supplementary Appendices: Preliminary Screening Survey of Metals and Dioxins in Fertilizers, Soil Amendments, and Soils in Washington State</u>

Appendix 1-E. Metals data for multiple and duplicate samples - 1998 sampling results.

Variations in product as well as analyses can be determined by comparing the results of two samples of several products collected independently. The analyses for Samples #1 and #2 of the Cozinco zinc micronutrient product gave similar results. Note that these samples were obtained from different sources and are not duplicate samples. Although the average RPD for detected pairs of like metals was 57%, the results for each metal agreed within 5.4 mg/kg-dw. Analyses for total metals were expressed in terms of dry weight (dw) so that the concentration of metals reported represents the concentration in a dried sample. The two Fort James NutriLime sample results were also similar, with an average relative percent difference of 24% for detected pairs of like metals. The two samples of Frit F-503G gave divergent results, with an average RPD of 78%. Cadmium, lead, and silver concentrations for those samples differed considerably.

Multiple Samples	As	Ba	Cd	Cr	Pb	Hg	Se	Ag	
Abbreviated Product Description	mg/Kg - dw	mg/Kg - dw	mg/Kg - dw	mg/Kg - dw	mg/Kg - dw	mg/Kg - dw	mg/Kg - dw	mg/Kg - dw	Lab Log#
Fort James Nutri Lime 08/98	28.5	543	1.44	39	89.3	0.158	0 3 U	0.444	338181
Fort James Nutri Lime 10/97			0.93	47.5	92.3	0.210	0 3 UJ	0 3 U	448081
Cozinco Sample #1	0 3 U	0.37	10.2	7 6	51.5	0 005 U	0 3 U	3.0	318085
Cozinco Sample #2	0 3 U	0.13	5.0	2.2	52.2	0 005 U	0 3 U	2 7	338201
Frit F-503G Sample #1	21.7	124	10 9	184	588	8.22	1.0	2.0	318086
Frit F-503G Sample #2	32.6	137	92 0	251	3490	11.9	0.45	9.27	348214

Duplicate Samples	As	Ba	Cd	Cr	Pb	Hg	Se	Ag	Lab
Abbreviated Product Description	mg/Kg - dw	mg/Kg - dw	mg/Kg - dw	mg/Kg - dw	mg/Kg - dw	mg/Kg - dw	mg/Kg - dw	mg/Kg - dw	Log#
Cozinco Sample #2	0 3 U	0.13	5.0	2.2	52.2	0 005 U	0 3 U	2.7	338201
Cozinco Sample #2 duplicate	0 3 U	0 11	4 9	1.9	53.9	0 005 U	0 3 U	2 6	338202
Hydro-Agri/Viking Ship FS/31	0 3 U	2.86	0 03 U	6.95	35.3	0 005 U	0 3 U	0 05 U	328139
Hydro-Agri/Viking Ship FS/31 dupe	0 3 U	3.07	0 03 U	6.9	37.7	0 005 U	0 3 U	0 05 U	328125
Webfoot Rhododendron	1 8 J	19.0 J	70.4	612 J	12	0.0077	0 3 UJ	0 05 U	328140
Webfoot Rhododendron duplicate	1.1	13 7 J	69.4	844 J	6.20 J	0.0064	0 3 UJ	0.061	328126
Whitney Farms 100% Organic Citrus	0.96	40.9	0 497	4.5	1.5	0 005 U	1.1	0 05 U	328138
Whitney Farms 100% Organic dupl	0.75	36.7	0.443	5.12	1.6	0.0055	0.73	0 05 U	328127
Evergro 23-3-23	0.90	4.16	5.98	24.5	10.4	0.026	0 3 U	0 05 U	338188
Evergro 23-3-23 duplicate	0.80	3.25	5.95	24.5	35.2	0 005 U	0 3 U	0 05 U	338189
NuLife Agro 10-15-10	1.8	12 7	58 9	175	52.4	0.0745	0 3 U	0 05 U	338195
NuLife Agro 10-15-10 duplicate	2.0	8.66	47 1	138	22.3	0.0928	0 3 U	0 05 U	338196

bold detected value

U - The analyte was not detected at or above the reported result

J - The analyte was postively identified The associated numerical result is an estimate

UJ - The analyte was not detected at or above the reported estimated result

Appendix 1-F - Dioxin data for multiple and duplicate samples - 1998 sampling results.

Multiple Samples *Abbreviated Product Description*	TEQ ND=0 pptr*	TEQ ND=1/2DL pptr*	TEQ ND=DL pptr*
Fort James Nutri Lime (two sample times)			
sampled 10/20/97	35 4	35 8	36 1
sampled 08/10/98	7 35	7 39	7.42
Cozinco 35 50% zinc (two distributors)			
Sample #1 (sampled 7/31/98)	ND	1 13	2 27
Sample #2 (sampled 8/14/98)	0 07	1 12	2 17
Frit F-503G (two distributors)			
Sample #1 (sampled 8/17/98)	26 8	28 8	30 8
Sample #2 (sampled 8/31/98)	145	148	152

Duplicate Samples

Abbreviated Product Description			
Cozinco 35 50% zinc			
Sample #2	0 07	1 12	2.17
Sample #2 duplicate	0 06	0 67	1 28
Hydro-Agri/Viking Ship FS/31			
Sample	ND	0 81	1 63
Sample duplicate	<0 01	0 54	1 08
Webfoot Rhododendron Food			
Sample	0 01	1 04	2 06
Sample duplicate	0 35	1 19	2.03
Whitney Farms Citrus, Berry, and Vine			
Sample	0 01	1 92	3 83
Sample duplicate	0 01	1 64	3 28
Evergro 23-3-23			
Sample	ND	1.37	2 73
Sample duplicate	ND	1 71	3 43
NuLife 10-15-10			
Sample	ND	2 52	5 03
Sample duplicate	0 02	3 69	7.37

ND = non-detect   < = less than

Appendix 1-G. Total metals in soil amendments, fertilizers, and micronutrients - 1998 sampling results.

Soil Amendments

	As	Ba	Cd	Cr	Pb	Hg	Se	Ag	Lab Log#
Abbreviated Product Description	mg/kg-dw	mg/kg-dw	mg/kg-dw	mg/kg-dw	mg/kg-dw	mg/kg-dw	mg/kg-dw	mg/kg-dw	
Ponderay Newsprint Fiberay SC	**0.45**	**53.0**	**0.586**	**6.3**	**2.73**	**0.044**	0.3 U	0.05 U	318082

Bulk/Packaged Agricultural Fertilizers

	As	Ba	Cd	Cr	Pb	Hg	Se	Ag	Lab Log#
Abbreviated Product Description	mg/Kg-dw	mg/Kg-dw	mg/Kg-dw	mg/Kg-dw	mg/Kg-dw	mg/Kg-dw	mg/Kg-dw	mg/Kg-dw	
Northwest Alloys High-Mag Gro	0.3 U	**27.6**	0.03 U	**1.7**	**4.21**	0.005 U	0.3 U	**1.1**	318081
Fort James Nutri Lime	**28.5**	**543**	**1.44**	**39**	**89.3**	**0.158**	0.3 U	**0.444**	338181
Unocal Ammonium Nitrate	0.3 U	**0.83**	**0.042**	0.5 U	0.74	0.005 U	0.3 U	0.05 U	318084
Agrium Ammonium Phosphate Sulfate	**0.60**	**5.52**	**160**	**196**	**2.90**	**0.019**	0.3 U	0.05 U	328131
IMC Kalium Potash	0.3 U	0.1 U	0.03 U	0.5 U	**0.28 J**	0.005 U	0.3 U	0.05 U	338184
Kelly Green Recycled Fresh Fish	**1.9**	0.1 U	**4.3**	**13.0**	0.2 U	**0.0768**	**0.32**	0.05 U	338182
UAP 0-45-0	**1.0**	**8.01**	**106**	**378**	**3.19**	0.005 U	0.3 U	0.05 U	348210
High Yield Sulfur	0.3 U	**1.21**	0.03 U	0.5 U	**0.40**	0.005 U	0.3 U	0.05 U	348207

Agricultural Micronutrients

	As	Ba	Cd	Cr	Pb	Hg	Se	Ag	Lab Log#
Abbreviated Product Description	mg/kg-dw	mg/kg-dw	mg/kg-dw	mg/kg-dw	mg/kg-dw	mg/kg-dw	mg/kg-dw	mg/kg-dw	
Frit F-503G Sample #1	**21.7**	**124**	**10.9**	**184**	**588**	**8.22**	**1.0**	**2.0**	318086
Frit F-503G Sample #2	**32.6**	**137**	**92.0**	**251**	**3490**	**11.9**	**0.45**	**9.27**	348214
Frit F-420G *	**40.1**	**181**	**135**	**1060**	**9490**	**1.09**	**5.60**	**20.6**	318087
RSA Ruffin-Ready Zn	0.3 U	**0.60**	**0.095**	0.5 U	**0.42**	0.005 U	0.3 U	0.05 U	318083
Tech-Flo Zeta Zinc 22	0.3 U	**0.68 J**	**1.9**	**3.8**	**8.92**	0.005 U	0.3 U	**3.2**	328132
Green Label Super Starter	**0.57**	**18.1**	**11.1**	**56.3**	**19.0**	0.005 U	0.3 U	**0.071**	338185
Cozinco Sample #1	0.3 U	**0.37**	**10.2**	**7.6**	**51.5**	0.005 U	0.3 U	**3.0**	318085
Cozinco Sample #2	0.3 U	**0.13**	**5.0**	**2.2**	**52.2**	0.005 U	0.3 U	**2.7**	338201
Bioplus Micro 700	**2.5**	**0.30**	**0.676**	0.5 U	0.2 U	0.005 U	0.3 U	0.05 U	328133
Horizon Ag Micro-Plus	**0.56**	**0.27**	**0.34**	**0.53**	**4.14**	0.005 U	**0.37**	0.05 U	328135
Western Farm/Monteray 9% Zn	0.3 U	0.1 U	**0.13**	0.5 U	**1.8**	**0.0068**	**0.34**	0.05 U	328134
Monteray 10% Zinc	0.3 U	**0.17**	**1.6**	1 U	**0.32**	0.005 U	0.3 U	0.05 U	338186
Hydro-Agri/Viking Ship FS/31	0.3 U	**2.86**	0.03 U	**6.95**	**35.3**	0.005 U	0.3 U	0.05 U	328139

* The sample of this material was collected in Oregon. The product is not registered or sold in Washington.

Appendix 1-G - (cont'd) - 1998 Sampling Results

Home-Use Packaged Fertilizer Products

Abbreviated Product Description	As mg/kg-dw	Ba mg/kg-dw	Cd mg/kg-dw	Cr mg/kg-dw	Pb mg/kg-dw	Hg mg/kg-dw	Se mg/kg-dw	Ag mg/kg-dw	Lab Log#
J.R. Simplot Best 6-20-20XB	**3.03**	**9.96**	**43.5**	**189**	**85.6**	0 005 U	0 3 U	0 05 U	338198
Gaia's Own Cottonseed Meal	**1.0**	**25.6**	0 03 U	0 5 U	**2550**	0 005 U	0 3 U	**0.051**	338183
A H Hoffman Ace Tomato & Veg	**1.4**	**20.6**	**1.24**	**139**	**4.16**	**0.0555**	0 3 U	0 05 U	348209
Fred Meyer Moss Control	**0.62**	**1.58**	**9.83**	**28.6**	**5.97**	0 005 U	0 3 U	0 05 U	348211
Winter Green 15-10-25	**1.7**	**1.45**	**26.9**	**106**	**2.67**	0 005 U	0 3 U	0 05 U	338197
Webfoot Turf Treat 15-5-10	**1.6**	**13.8**	**15.7**	**52.1**	**28.3**	**0.257**	**0.46**	**0.19**	348213
Terosa Rose Food	**7.05**	**25.1**	**30.1**	**103**	**8.03**	**1.13**	**2.6**	**0.567**	328146
Evergro 23-3-23	**0.90**	**4.16**	**5.98**	**24.5**	**10.4**	**0.026**	0 3 U	0 05 U	338188
Pursell Sta-Green Azalea	**1.4**	**14.5**	**1.78**	**29.9**	**76.0**	**0.364**	0 3 U	0 05 U	338200
Schultz Bloom Plus	**0.58**	**1.15**	0 03 U	**11.2**	0 2 U	**0.045**	0 3 U	0 05 U	328143
TurfGo 12-0-0	0 3 U	**0.49**	**0.048**	**2.6**	**0.42**	0 005 U	0 3 U	0 05 U	338191
Pace NuLife 10-20-20	**1.3**	**3.44**	**89.3**	**254**	**14.8**	**0.0865**	0 3 U	0 05 U	338194
Liquinox Iron and Zinc	0 3 U	**0.14**	0 03 U	0 5 U	**0.22**	0 005 U	0 3 U	0 05 U	328142
McLendon Weed and Feed 15-5-5	**1.6**	**59.5**	**15.2**	**5060**	**10.9**	**0.017**	0 3 U	0 05 U	338190
NuLife Agro 10-15-10	**1.8**	**12.7**	**58.9**	**175**	**52.4**	**0.0745**	0 3 U	0 05 U	338195
Pursell Sta-Green Nursery Special	**1.3**	**7.27**	**2.5**	**26.0**	**32.6**	**0.652**	0 3 U	**0.16**	348206
QC 30% Iron	**0.3 U**	**2.26**	**2.8**	**5.77**	**16**	0 005 U	0 3 U	0 05 U	348212
Ringer/Amturf Wildflower Mix	**0.34**	**26.0**	**0.161**	**4.9**	**1.5**	**0.014**	0 3 U	0 05 U	328137
Schultz Soluble for Orchids	0 3 U	**1.14**	0 03 U	**4.4**	**0.35 J**	0 005 U	0 3 U	0 05 U	338204
Osmocote Vegetable and Bedding	0 3 U	**141**	0 03 U	**4.8**	**1.2**	0 005 U	0 3 U	0 05 U	328145
Peters Professional All-Purpose	**0.33**	**1.00**	0 03 U	**3.0**	0 2 U	0 005 U	0 3 U	0 05 U	338203
Thrifty Pay-Less Tomato & Veg.	**1.9 J**	**23.6**	**62.1**	**231**	**6.58**	**0.139**	**5.71 J**	**2.88**	338205
Whitney Farms 100% Organic Citrus	**0.96**	**40.9**	**0.497**	**4.5**	**1.5**	0 005 U	**1.1**	0 05 U	328138
Ortho Upstart	0 3 U	**0.15**	0 03 U	**1.2**	**0.22**	0 005 U	0 3 U	0 05 U	338193
NuLife All-Purpose Trace Elements	**75.2**	**205**	**45.9**	**417**	**1940**	**0.206**	**1.9**	**5.28**	328144
Whitney Farms Jersey Green Sand	**11.4**	**16.0**	**0.12**	**79.2**	**4.26**	**0.0054**	**0.94**	0 05 U	338199
Whitney Farms Iron Sulfate	0 3 U	**2.0**	0 03 U	**17**	**29.8**	0 005 U	0 3 U	0 05 U	328141
Hydro-Feed with Polyon 20-10-10	**1.4**	**3.04**	**18.9**	**72.4**	**434**	**0.103**	0 3 U	0 05 U	338187
Hi-Yield Pecan and Fruit Tree Fertilizer	**1.1**	**43.7**	**1.30**	**37.8**	**2.26**	0 005 U	0 3 U	0 05 U	348208
Webfoot Rhododendron	1 8 J	**19.0 J**	**70.4**	**612 J**	**12**	**0.0077**	0 3 UJ	0 05 U	328140
Ringer Magic Start Grass Patch	0 3 U	**7.96**	**0.189**	**15.3**	**17**	0 005 U	0 3 U	0 05 U	338192

bold detected value
U - The analyte was not detected at or above the reported result.
J - The analyte was postively identified. The associated numerical result is an estimate.
UJ - The analyte was not detected at or above the reported estimated result

Appendix 1-H. TCLP limits.

Metal	TCLP Limit, mg/L	20X TCLP Limit mg/kg dw
Arsenic	5.0	100
Barium	100.0	2,000
Cadmium	1.0	20
Chromium	5.0	100
Lead	5.0	100
Mercury	0.2	4.0
Selenium	1.0	20
Silver	5.0	100

TCLP analyses were run for samples with total metals concentrations greater than 20X TCLP limit. When total metals are below 20X the TCLP limit, TCLP is, by definition, not exceeded.

Appendix 1-I. Comparison of January and July-August metals in fertilizer products - 1998 sampling results.

Abbreviated Product Description	As	Ba	Cd	Cr	Pb	Hg	Se	Ag	Lab Log#
	mg/kg-dw	mg/kg-dw	mg/kg-dw	mg/kg-dw	mg/kg-dw	mg/kg-dw	mg/kg-dw	mg/kg-dw	
Kelly Green Fresh Fish Fert 8/98	**1.9**	0 1 U	**4.3**	**13.0**	0 2 U	**0.0768**	**0.32**	0 05 U	328182
Kelly Green Fresh Fish Fert 1/98	**2 U**	**0.14**	**4.20**	**20.2**	1 U	**0.111**	2 U	0 25 U	
Cozinco Sample #1 7/98	0 3 U	**0.37**	**10.2**	**7.6**	**51.5**	0 005 U	0 3 U	**3.0**	318085
Cozinco Sample #2 8/98	0 3 U	**0.13**	**5.0**	**2.2**	**52.2**	0 005 U	0 3 U	**2.7**	338201
Cozinco 1/98	4 U	**0.51**	**10.3**	**2.1**	**119**	0 005 U	4 U	**2.9**	058156
QC 30% Iron 8/98	0 3 U	**2 26**	**2.8**	**5.77**	**16**	0 005 U	0 3 U	0 05 U	
QC 30% Iron 1/98	100 UJ	**2.1**	**2.1**	**6.18**	**23.1**	0 005 U	200 UJ	2 UJ	058172

bold detected value

U - The analyte was not detected at or above the reported result.

J - The analyte was postively identified. The associated numerical result is an estimate.

UJ - The analyte was not detected at or above the reported estimated result.

Appendix 1-J. Fertilizer metals data - January 1998 samples.*

Lab Log#	058155	058156	058157	058158	058159	058160	058161	058162
Field ID.	H0939	H1924	H0762	H1255	H1253	H1251	H1257	H0950
	mg/L**	mg/kg-dw	mg/L**	mg/kg-dw	mg/kg-dw	mg/kg-dw	mg/kg-dw	mg/kg-dw
Aluminum	3 7	82 6	1 2 U	795	16	4 2	95 8	2 U
Antimony	2 7 U	4 U	2 5 U	4 U	4 U	8 2	4 U	4 U
Arsenic	2 7 U	4 U	2 5 U	4 U	4 U	4 U	4 U	4 2
Barium	0 07 U	0 51	0 065	31 5	85 5	0 5	3 41	2 44
Beryllium	0 07 U	0 1 U	0 062 U	0 19	0 1 U	0 1 U	0 1 U	0 1 U
Cadmium	1 5	10 3	0 31 U	0 5 U	0 5 U	8 19	0 5 U	0 5 U
Calcium	26 5	299	8 7	1190	190000	657	32000	845
Chromium	0 34 U	2 1	0 31 U	0 5 U	10 5	1 2	1 4	1.1
Cobalt	0 35	0 5 U	0 31 U	0 5 U	0 5 U	0 5 U	0 5 U	288
Copper	2 2	3 8	0 62 U	1 U	1 U	3 9	15 5	15400
Iron	1 4 U	2130	6 0	430	11	29 4	524	35500
Lead	1 4 U	119	1 2 U	3 5	2 U	13	2 0	2 U
Magnesium	3 4 U	50 3	3 1 U	218	87 1	261	4950	51000
Manganese	3 10	291	0 28	42 9	3 27	10.2	15.4	36400
Molybdenum	0 34 U	0 5 U	0 43	0 5 U	0 5 U	0.5 U	0 5 U	846
Nickel	60 7	4 3	0 62 U	1 1	1 U	8 2	1 6	1 5
Potassium	1400	100 U	2020	140	100 U	580	45300	1680
Selenium	2 7 U	4 U	2 5 U	4 U	4 U	4 U	4 U	4 U
Silver	2 0	2 9	0 31 U	0 5 U	0 5 U	2 7	0 5 U	0 76
Sodium	843	502	3430	1270	128	11600	4210	23400
Strontium	0 068 U	0 92	0 11	26 4	3050 J	4 82	132 J	11 3 J
Thallium	3 4 U	5 U	3 1 U	5 U	5 U	5 U	5 U	5 U
Titanium	0 34 U	0 57	0 31 U	17	4.4	0 5 U	4 5	55 8
Vanadium	0 14 U	0 2 U	0 12 U	0 2 U	0 2 U	0 2 U	0 37	0 2 U
Zinc	258000	348000	1 29	13	15	339000	64 8	13900
Mercury	3 3**	0 005 U	3 1** U	0 005 U	0 005 U	0.005 U	0.077	0 005 U

* Fertilizers are listed in Appendix 1-M.

** Liquid samples Mercury as ug/L for these samples

Appendix 1-J - (cont'd) - January 1998 samples.

Lab Log	058163	058164	058165	058166	058167	058168	058169	058170
Field ID	H0761	H1256	G3682	G3683	G3684	G3685	G3687	G3688
	mg/kg-dw	mg/kg-dw	mg/L**	mg/kg-dw	mg/kg-dw	mg/kg-dw	mg/kg-dw	mg/kg-dw
Aluminum	2 U	1530	346	751	126	7 2	5500	186
Antimony	4 U	4 U	2 U	5 4	4 U	4 U	4 U	4 U
Arsenic	4 U	4 U	2 U	4 U	4 U	4 U	14	20
Barium	0 94	9.87	0 14	11 5	0.84	0 16	53	7 56
Beryllium	0 1 U	0 15	0 19	0 1 U	0 1 U	0 1 U	4 2	0 1 U
Cadmium	2 5	0 5 U	4 20	0 5 U	0 5 U	0 5 U	115	0 5 U
Calcium	753	232000	551	267000	399000	111	289000	30100
Chromium	3 9	11 5	20 2	11 5	0 5 UJ	0.5 U	454	1 1
Cobalt	64 6	0 5 U	0 25 U	0 95	0 5 U	0 5 U	1 6	0 96
Copper	5 U	1.6	2.0	15 6	6.4	3 7	79 0	3 2
Iron	2 U	893	251	2310	209	51 2	5300	433
Lead	2 U	2 U	1 U	7 0	2 U	2 4	8 1	2 U
Magnesium	533	18400	262	478	1710	17	2750	8020
Manganese	303000	39 5	20 2	46 8	28 3	0 92	86 6	34 2
Molybdenum	34 5	0 5 U	0 56	0 5 U	0 5 U	0 5 U	16 1	0 58
Nickel	66 2	1.1	5 1	2 7	1 3	1 U	119	1 4
Potassium	2690	510	2160	150	100 U	100 U	3420	20000
Selenium	4 U	4 U	2 U	4 U	4 U	4 U	27	4 U
Silver	17 9	0 5 U	0.25 U	0 5 U	0 5 U	0 5 U	4 9	0 5 U
Sodium	681	1170	1290	98 2	27	23	3370	26200
Strontium	16	883 J	2 77	1130 J	838	1 86	752 J	605 J
Thallium	5 U	5 U	2.5 U	5 U	5 U	5 U	5 U	5 U
Titanium	0.5 U	18 2	4.70	67 2	0 5 U	0 5 U	56	6 75
Vanadium	0 2 U	0.98	35 9	2 77	0 4	0 2 U	848	2 32
Zinc	430	11	70 5	15	4 4	6 7	1320	34 7
Mercury	0 005 U	0 005 U	111**	0 005 U	0 005 U	1 25 J	0 315	0 024

** Liquid samples Mercury as ug/L for these samples

Appendix 1-J - (cont'd) - January 1998 samples.

Lab Log	058171	058172	058173	058174	058175	058176	058177
Field ID	G3689	G3489	G3490	G3491	G3492	H0947	G3690
	mg/kg-dw	mg/kg-dw	mg/kg-dw	mg/kg-dw	mg/kg-dw	mg/kg-dw	mg/kg-dw
Aluminum	13000 J	134	538	5110	23 3	6 9	12600
Antimony	4 U	4 U	4 U	4 U	4 1	4 U	22 J
Arsenic	7.0	100 UJ	4 U	15	4 U	4 U	4460
Barium	13 8	2 1	14	194	0 42	1 81	17 1 J
Beryllium	1 79	0 1 U	0 18	0 13	0 1 U	0 1 U	0 22
Cadmium	2 0	2 1	0 70	2 3	0 5 U	0 5 U	18 9
Calcium	177000 J	4870	20100	15300	11600	226	33700
Chromium	59 4 J	6 18	5 11	339	10	0 59	17 2
Cobalt	2 5	49 5	0 5 U	3.6	0 5 U	0 5 U	12 6
Copper	2.0	25	50 8	229	4 0	1.1	289
Iron	11900	299000	957	37800	101	32 7	112000
Lead	2 U	23 1	5 8	64 2	2 U	2 U	2420
Magnesium	14200 J	9090	7470	4530	110	51 3	18400
Manganese	208 J	1050	54 2	210	16 9	5 31	722
Molybdenum	3 9	0 5 UJ	1 5	7 63	0 5 U	0 5 U	4 7
Nickel	8 8	60	3 1	31	1 1	1 9	9 4
Potassium	2630	100 U	113000	5060	100	6480	180
Selenium	4 U	200 UJ	4 U	4 UJ	4 U	4 U	4 UJ
Silver	0 5 U	2 UJ	0 5 U	13 2	0 5 U	0 5 U	16
Sodium	1150	163	1230	1270	33	103000	58.1
Strontium	272 0	1 91	45 9	232	4 95	2 65 J	37 5 J
Thallium	5 U	50 UJ	5 U	7 4	5 U	5 U	13
Titanium	176	2250	11 5	44 8	0 91	0 5 U	53 J
Vanadium	92	30	6 82	8 57	0 2 U	0 2 U	40 0
Zinc	43	291	96 5	477	4 6	11	8760
Mercury	0 005 U	0.005 U	0 005 U	0 778	0 005 U	0 005 U	13.7

Appendix 1-K. Fertilizer products January 1998 samples.

Lab Log #	Field I.D.	Material	Company
058155	H0939	Zinc	UAP Northwest
058156	H1924	Granular zinc sulfate monohydr	Cozinco
058157	H0762	Trisert CB	Tessenderlo-Kerley
058158	H1255	Sulfur	Montana Sulfur
058159	H1253	Calcium nitrate	Hydro-Agri
058160	H1251	Zinc sulfate monohydrate	Chemical and Pigment Company
058161	H1257	Organic turf fertilizer	BioProducts
058162	H0950	Microplex	Miller Chemical Company
058163	H0761	Manganese	American Microtrace
058164	H1256	Gypsum	Greenacres Gypsum
058165	G3682	Organic fish fertilizer	Global Recycling
058166	G3683	Gypsum	U.S. Gypsum
058167	G3684	Limestone	Chemical Lime of Canada
058168	G3685	Ammonium sulfate	Agrium
058169	G3687	Rock phosphate	Garden Grow Company
058170	G3688	Kelp meal	Garden Grow Company
058171	G3689	Super phosphate	Voluntary Purchasing Group
058172	G3489	Ferrous sulfate	QC Corporation
058173	G3490	Organic turf fertlizer	Ecosoil Systems
058174	G3491	Biosolid fertilizer	Milorganite
158175	G3492	Diammonium phosphate	Monsanto
058176	H0947	Solubor	U.S Borax
058177	G3690	Ironite	Ironite Company

Appendix 1-L. Metals results for waste-derived fertilizers and soil amendments - October 1997.

Source	Product	As	Cd	Cu	Pb	Hg	Ni	Zn
Fort James	hog fuel boiler ash							
	Dioxin study	49 3	0 93	91 5J	92 3	0 210	33 1	381J
	Metals screening study	66	2 1U	159	171	0 414	32	581
Holnam	cement kiln dust							
	Dioxin study	18 8	2 4	172J	230	0 772	19 4	1380J
	Metals screening study	37	3 6	158	150	0 041	18	1770
Bay Zinc	Zinc micronutrient							
	18% Blu-Min							
	Dioxin study	27 0	269	1730J	11,700	4 32	70 0	184000J
	Dioxin study duplicate	28 1	267	1750J	11,600	4 56	68 8	189,000J
	Metals screening study	34U	275J	1680J	11,300J	3 36J	83J	178,000J
	LHM							
	Dioxin study	29 5	21 5	419J	738	1 89	33 2	225000J
	Metals screening study	35U	52 1	672	1400		61 6	203,000
	*Liquid product**							
	Dioxin study	46 2J*	25400*	9020*	13300*	20 5*	3570*	81000000*

Dioxin study - Ecology sampling of waste-derived fertilizer products and micronutrients, (unpublished)

Metals screening study - Department of Agriculure sampling of fertilizers and industrial by-product fertilizers (Bowhay, et al, 1997)

* Liquid product as µg/L Liquid density = 1 33

J - estimated value

U - The analyte was not detected at or above the reported result

Appendix 1-L - (cont'd) - October 1997.

Source	Product	Sb	Be	Cr	Se	Ag	Tl
Fort James	<u>hog fuel boiler ash</u>						
	Dioxin study	5UJ	0 65	47 5J	0 3UJ	0 3U	0 3UJ
	Metals screening study	21UJ	0 7U	46 7	27U	2 1UJ	27U
Holnam	<u>cement kiln dust</u>						
	Dioxin study	5UJ	0 31	29 8J	0 3UJ	0 97	0 3UJ
	Metals screening study	30UJ	1U	73 2	40U	3UJ	40U
Bay Zinc	<u>Zinc micronutrient</u>						
	18% Blu-Min						
	Dioxin study	30UJ	0 32	529J	7 88J	37 4	0 3UJ
	Dioxin study duplicate	30UJ	0 34	525J	7 90J	38 1	0 3UJ
	Metals screening study	42J	1 1U	580J	45U	38 5J	45U
	LHM						
	Dioxin study	30UJ	0 14	29 9J	2 2J	2 6	0 3UJ
	Metals screening study	44J	1 2U	67 8	50U	5 4	100U
	Liquid product*						
	Dioxin study	10,000UJ*	168J*	3170*	62 3J*	735*	6UJ*

* Liquid product as µg/L Liquid density = 1 33

fall 1997

J - estimated value

U - The analyte was not detected at or above the reported result

Appendix 1-M. Dioxin TEQs in soil amendments, fertilizers, and micronutrients – 1998 sampling results.

Soil Amendments

Abbreviated Product Description	TEQ ND=0 pptr*	TEQ ND=1/2DL pptr*	TEQ ND=DL pptr*	*Lab Log#*
Ponderay Newsprint Fiberay SC	0.40	6.00	11.6	318082

Bulk/Packaged Agricultural Fertilizers

Abbreviated Product Description	TEQ ND=0 pptr*	TEQ ND=1/2DL pptr*	TEQ ND=DL pptr*	*Lab Log#*
Northwest Alloys High-Mag Gro	0.01	0.46	0.91	318081
Fort James Nutri Lime	7.35	7.39	7.42	338181
Unocal Ammonium Nitrate	0.00	2.05	4.09	318084
Agrium Ammonium Phosphate Sulfate	0.00	1.08	2.15	328131
IMC Kalium Potash	0.02	0.73	1.45	338184
Kelly Green Recycled Fresh Fish	1.35	5.15	9.37	338182
UAP 0-45-0	0.05	0.49	0.93	348210
High Yield Sulfur	0.00	2.05	4.10	348207

Agricultural Products with Micronutrients

Abbreviated Product Description	TEQ ND=0 pptr*	TEQ ND=1/2DL pptr*	TEQ ND=DL pptr*	*Lab Log#*
Frit F-503G Sample #1	26.8	28.8	30.8	318086
Frit F-503G Sample #2	145	148	152	348214
Frit F-420G **	287	287	287	318087
RSA Ruffin-Ready Zn	<0.01	0.49	0.98	318083
Tech-Flo Zeta Zinc 22	0.01	0.74	1.48	328132
Green Label Super Starter	0.29	1.46	2.63	338185
Cozinco Sample #1	0.00	1.13	2.27	318085
Cozinco Sample #2	0.07	1.12	2.17	338201
Bioplus Micro 700	0.00	0.50	1.00	328133
Horizon Ag Micro-Plus	<0.01	1.66	3.31	328135
Western Farm/Monteray 9% Zn	<0.01	0.34	0.68	328134
Monteray 10% Zinc	0.04	0.54	1.03	338186
Hydro-Agri/Viking Ship FS/31	0.00	0.81	1.63	328139

* parts per trillion. Solid samples on dry-weight basis. Liquid samples on volume basis. TEQs with non-detects set at zero.

** The sample of this material was collected in Oregon. The product is not registered or sold in Washington.

Appendix 1-M - (cont'd) - 1998 sampling results.

Home-Use Packaged Fertilizer Products	TEQ ND=0	TEQ ND=1/2DL	TEQ ND=DL	*Lab Log#*
Abbreviated Product Description	pptr*	pptr*	pptr*	
J R Simplot Best 6-20-20XB	0 03	1 15	2 27	338198
Gaia's Own Cottonseed Meal	0 00	0 67	1 33	338183
A H Hoffman Ace Tomato & Veg.	0 06	1 41	2 76	348209
Fred Meyer Moss Control	0 03	1 02	2 02	348211
Winter Green 15-10-25	0 06	1 35	2 65	338197
Webfoot Turf Treat 15-5-10	1 20	2 10	3 01	348213
Terosa Rose Food	<0 01	1 44	2 88	328146
Evergro 23-3-23	0 00	1 37	2 73	338188
Pursell Sta-Green Azalea	0 15	1 75	3 34	338200
Schultz Bloom Plus	0 05	0 85	1 65	328143
TurfGo 12-0-0	0 03	0 54	1 05	338191
Pace NuLife 10-20-20	0 00	1 79	3 59	338194
Liquinox Iron and Zinc	<0 01	0 42	0.83	328142
McLendon Weed and Feed 15-5-5	5 42	6 62	7 83	338190
NuLife Agro 10-15-10	0 00	2 52	5.03	338195
Pursell Sta-Green Nursery Special	3 19	4 07	4 95	348206
QC 30% Iron	<0 01	1 03	2 06	348212
Ringer/Amturf Wildflower Mix	0 10	2 44	4 78	328137
Schultz Soluble for Orchids	0 09	0 90	1 71	338204
Osmocote Vegetable and Bedding	0 00	1 15	2 30	328145
Peters Professional All-Purpose	0 11	0 93	1 75	338203
Thrifty Pay-Less Tomato & Veg	0 01	2 38	4 74	338205
Whitney Farms 100% Organic Citrus	0 01	1 92	3 83	328138
Ortho Upstart	0 08	0 58	1 08	338193
NuLife All-Purpose Trace Elements	53 7	53 7	53 7	328144
Whitney Farms Jersey Green Sand	0 16	0 85	1 54	338199
Whitney Farms Iron Sulfate	<0.01	1 09	2 18	328141
Hydro-Feed with Polyon 20-10-10	0 00	1 25	2 51	338187
Hi-Yield Pecan and Fruit Tree Fertilizer	0 65	1 35	2 04	348208
Webfoot Rhododendron	0 01	1 04	2 06	328140
Ringer Magic Start Grass Patch	0 00	0 70	1 40	338192

* parts per trillion. Solid samples on dry-weight basis. Liquid samples on volume basis. TEQs with non-detects set at zero.
ND = non- detect DL = detection limit
ND = 0: if congener not detected, concentration assumed = 0
ND = 1/2 DL: if congener not detected, concentration assumed = 1/2 detect limit
ND = DL: if congener not detected, concentration assumed = detect limit

Appendix 1-N. Dioxin data and TEQ calculations – 1998 sampling results.

Appendix 1-N is included in a supplemental report, Ecology Publication 98-332:

Supplementary Appendices: Preliminary Screening Survey of Metals and Dioxins in Fertilizers, Soil Amendments, and Soils in Washington State

Appendix 1-O. Dioxin data and TEQ calculations - 1997 sampling results.

Appendix 1-O is included in a supplemental report, Ecology Publication 98-332

Supplementary Appendices: Preliminary Screening Survey of Metals and Dioxins in Fertilizers, Soil Amendments, and Soils in Washington State

Appendix 1-P. Dioxin TEQs in fertilizers and micronutrients -1997 sampling results

Bulk/Packaged Agricultural Fertilizers	TEQ ND=0 pptr**	TEQ ND=1/2DL pptr**	TEQ ND=DL pptr**	*Lab Log#*
Allied Minerals Dolomite	ND	0.42	0.84	448080
Fort James Nutri Lime	35.4	35.8	36.1	448081
Holnam Cement Kiln Dust	0 95	1 91	2 87	448083
Agricultural Micronutrients				
Bay Zinc K061	815	827	839	448084
Bay ZincTire Dust	1 62	2 70	3 78	448085
Bay Zinc Blu-Min	342	342	342	448087
Bay Zinc LHM	5 60	9 00	12 4	448088
Bay Zinc Liquid	0 64	0.64	0 64	448089

* Groupings by fertilizer type are tentative and may change
** Parts per trillion Solid samples on weight basis (ng/kg = pg/g) Liquid samples on volume basis (ng/L = pg/mL)
ND = non-detect DL = detection limit
ND = 0 if congener not detected, concentration assumed = 0
ND = 1/2 DL· if congener not detected, concentration assumed = 1/2 detect limit
ND = DL: if congener not detected, concentration assumed = detect limit

Appendices
for
2. Metals in Soils

Appendix 2-I. Analytical results of metals in soils study.

Lab #	Crop	Soil Type	pH	CEC meq	TOC-104°C %	TOC-70°C %	Phosph mg/Kg	As mg/Kg	Cd mg/Kg	Cu mg/Kg	Pb mg/Kg	Hg mg/Kg	Ni mg/Kg	Zn mg/Kg
Background Samples														
338710	NA	Warden silt loam	7 1	3 79	0 62	0 56	701	2 5	0 08	15 9	8 36	0 0065 J	12 2	56 2
338702	NA	Shano silt loam	7 7	3 46	0 68	0 61	616	2 5	0 084	14 6	5 47	0 008 J	14 1	38 7
358745	NA	Scoon silt loam	7 3	3 78	1 06	1 06	853	5 56	0 03 U	20 2	9 97	0 066 J	13 6	53 4
348729	NA	Novark silt loam	8 2	3 15	0 54	0 53	699	4 45	0 03 U	11 5	5 65	0 011 J	9 1	36 9
348730	NA	Kennewick silt loam	8 2	2 92	0 28	0 28	694	3 19	0 044	12 7	6 34	0 0047 J	11	38 2
358744	NA	Sagemoor silt loam	6 5	4 51	0 83	0 82	734	2 4	0 039	10 9	6 01	0 009 J	10	35 9
358746	NA	Ephrata fine sandy loam	7 8	3 77	0 76	0 76	671	3 14	0 98	13	9 21	0 012 J	10 4	46 5
358747	NA	Kennewick fine sandy loam	7 2	2 97	0 59	0 59	587	2 7	0 061	10 3	6 77	0 0045 J	9 5	32 5
348741	NA	Royal very fine sandy loam	6 4	3 93	0 46	0 46	805	3 51	0 062	14 6	8 21	0 008 J	10 1	47 9
358743	NA	Prosser very fine sandy loam	6 9	4 92	0 67	0 66	938	3 03	0 032	16 7	7 54	0 007 J	12 7	48 3
338712	NA	Quincy fine sand	7 4	3 3	0 21	0 19	987	2 4	0 042	13 1	5 76	0 0032 J	8 4	55 7
338726	NA	Quincy fine sand	7 7	2 21	0 33	0 32	873	3 81	0 042	9 89	6 12	0 004 UJ	8	46 1
338732	NA	Timmerman coarse sandy loam	7 6	3 16	0 3	0 27	1460	1 5	0 05	12 7	4 6	0 0032 J	8	52 7
Field Samples														
338701	Apples	Warden silt loam	6 1	3 32	0 53	0 5	618	2 3	0 21	14 5	8 23	0 009 J	13 6	53
338703	Apples	Shano silt loam	6	3 21	0 42	0 4	695	3	0 13	15 6	7 3	0 013 J	15 7	49 5
338705	Apples	Shano silt loam	6 3	3 21	0 55	0 49	712	2 6	0 15	14 4	7 16	0 008 J	14 9	53 3
338707	Alfalfa	Ephrata fine sandy loam	6 1	3 93	0 99	0 9	873	2 2	0 14	14 5	6 53	0 007 J	10 1	52 8
338709	Alfalfa	Warden silt loam	6 4	5 21	0 72	0 66	693	3 26	0 13	19	9 02	0 01 J	14 8	59 9
338711	Alfalfa	Quincy fine sand	6 7	2 52	0 33	0 3	944	3 23	0 069	10 7	6 81	0 003 UJ	8 1	57
338713	Bean	Timmerman coarse sandy loam	6 2	3 4	0 91	0 84	1050	2 1	0 092	13 4	5 78	0 0042 J	9 4	65
338715	Oats	Timmerman coarse sandy loam	6 6	3 49	0 84	0 77	819	2 5	0 089	13 3	6 21	0 0043 J	9 2	58 1
338717	Corn	Ephrata fine sandy loam/ *Malaga gravelly sandy loam*	5 5	3 91	1 14	1 05	656	2 5	0 13	13 4	6 14	0 005 J	9 2	53 2
338719	Alfalfa	Kennewick silt loam	7 8	3 53	0 56	0 52	750	3 9	0 09	13 3	6 56	0 011 J	11 2	45
338723	Alfalfa	Scoon silt loam	7	3 72	0 97	0 89	636	2 4	0 15	13 6	6 29	0 008 J	12 7	43 8
338725	Alfalfa	Quincy fine sand	6 7	2 56	0 41	0 37	852	3 67	0 088	9 49	5 94	0 003 UJ	7 9	51 4
338731	Alfalfa	Timmerman coarse sandy loam	7 6	3	0 44	0 41	1010	2 6	0 079	15 5	6 34	0 007 J	9 9	57 8
338733	Wheat	Kennewick silt loam	7 95	3 63	0 47	0 46	811 5	3 87	0 1	14 48	8 2	0 009 J	12 65	48 88
348728	Primrose	Novark silt loam	7 5	3 01	0 59	0 58	776	5 68	0 05	12 6	6 93	0 008 J	8 8	47 8
348737	Wheat	Kennewick silt loam	7 8	3 78	0 48	0 47	733	3 61	0 1	13 9	8 23	0 009 J	12 3	48 5
348738	Potato	Kennewick fine sandy loam	7 4	3 5	1 06	1 06	714	4 51	0 059	13 4	7 7	0 006 J	12 3	43 6
348739	Sugar	Sagemoor silt loam	7 9	4 39	0 6	0 59	1060	4 48	0 051	17 7	9 59	0 009 J	11 8	59 2
348740	Bean	Royal very fine sandy loam	7 6	5 33	0 48	0 48	974	5 39	0 086	17 7	8 95	0 01 J	10 5	59
359742	Pasture	Prosser very fine sandy loam	7 7	4 05	0 89	0 89	696	3 16	0 081	15 7	7 66	0 008 J	10 7	55 2
348733R	Wheat	Kennewick silt loam	7 9	3 74	0 48	0 47	796	3 86	0 12	14 2	8 06	0 0089 J	13	48
348734R	Wheat	Kennewick silt loam	8	3 77	0 45	0 45	834	3 91	0 13	14 6	8 5	0 011 J	13 2	49 4
348735R	Wheat	Kennewick silt loam	8	3 55	0 46	0 46	827	3 87	0 15	14 7	8 48	0 0088 J	12 1	50
348736R	Wheat	Kennewick silt loam	7 9	3 45	0 47	0 47	789	3 82	0 10	14 4	7 77	0 0081 J	12 3	48 1

R = Replicate sample
U = The analyte was not detected at or above the reported result
J = The analyte was positively identified the associated numerical result is an estimate
UJ = The analyte was not detected at or above the reported estimated result

Appendix 2-I. Analytical results of metals in soils study (continued).

Lab #	Crop	Soil Type	As - DTPA	Cd - DTPA	Cu - DTPA	Pb - DTPA	Zn - DTPA
Background Samples							
338710	NA	Warden silt loam	0 48 U	0 08	4 71	1 36	3 64
338702	NA	Shano silt loam	0 48 U	0 04 U	2 76	0 28	0 52
358745	NA	Scoon silt loam	0 48 U	0 044	2 25	0 89	1 86
348729	NA	Novark silt loam	0 48 U	0 04 U	2 82	0 57	1 03
348730	NA	Kennewick silt loam	0 53	0 04 U	2 47	0 42	0 4
358744	NA	Sagemoor silt loam	0 48 U	0 04 U	2 12	0 69	1 25
358746	NA	Ephrata fine sandy loam	0 48 U	0 04 U	2 8	1 07	1 93
358747	NA	Kennewick fine sandy loam	0 48 U	0 04 U	0 072	0 16 U	0 067
348741	NA	Royal very fine sandy loam	0 48 U	0 052	2 69	0 53	1 47
358743	NA	Prosser very fine sandy loam	0 48 U	0 04 U	4 69	0 78	0 74
338712	NA	Quincy fine sand	0 48 U	0 04 U	1 75	0 6	3 05
338726	NA	Quincy fine sand	0 48 U	0 04 U	1 13	0 63	0 958
338732	NA	Timmerman coarse sandy loam	0 48 U	0 04 U	1 92	0 29	0 49
Field Samples							
338701	Apples	Warden silt loam	0 48 U	0 13	2 89	1 52	6 12
338703	Apples	Shano silt loam	0 48 U	0 063	2 73	0 72	3
338705	Apples	Shano silt loam	0 48 U	0 08	3 53	1 15	4 2
338707	Alfalfa	Ephrata fine sandy loam	0 48 U	0 089	2 36	0 86	6 2
338709	Alfalfa	Warden silt loam	0 48 U	0 04 U	2 01	0 58	1 83
338711	Alfalfa	Quincy fine sand	0 48 U	0 04 U	1 97	0 23	0 32
338713	Bean	Timmerman coarse sandy loam	0 48 U	0 057	2 21	0 44	5 02
338715	Oats	Timmerman coarse sandy loam	0 48 U	0 052	2 35	0 6	3 33
338717	Corn	Ephrata fine sandy loam/ Malaga gravelly sandy loam	0 48 U	0 105	2 47	0 55	3 69
338719	Alfalfa	Kennewick silt loam	0 48 U	0 056	3 13	0 76	3 99
338723	Alfalfa	Scoon silt loam	0 56	0 094	3 31	1 08	3 36
338725	Alfalfa	Quincy fine sand	0 48 U	0 04U U	1 43	0 57	3 29
338731	Alfalfa	Timmerman coarse sandy loam	0 49	0 068	3 42	0 58	3 58
338733	wheat	Kennewick silt loam	0 49	0 09	3 38	1 15	4 93
348728	Primrose	Novark silt loam	0 62	0 048	2 48	0 88	4 2
348737	Wheat	Kennewick silt loam	0 54	0 058	4 22	0 74	5 31
348738	Potato	Kennewick fine sandy loam	0 6	0 051	2 57	0 965	3
348739	Sugar	Sagemoor silt loam	0 48	0 04 U	2 59	0 39	1 19
348740	Bean	Royal very fine sandy loam	0 74	0 113	4 85	1 31	6 97
358742	Pasture	Prosser very fine sandy loam	0 48 U	0 084	3 19	0 86	4 67
348733R	Wheat	Kennewick silt loam	0 48 U	0 095	3 34	1 11	4 92
348734R	Wheat	Kennewick silt loam	0 48 U	0 093	3 44	1 12	5 04
348735R	Wheat	Kennewick silt loam	0 53	0 104	3 56 J	1 16	5 22 J
348736R	Wheat	Kennewick silt loam	0 48 U	0 08	3 16	1 19	4 53

R = Replicate sample
U = The analyte was not detected at or above the reported result
J = The analyte was positively identified, the associated numerical result is an estimate
UJ = The analyte was not detected at or above the reported estimated result

Procedural Blanks

The procedural blanks associated with these samples showed no analytically significant levels of analyte except zinc Zinc was present in two of the three ICP procedure blanks Sample zinc results were greater than ten times the blank results, so the data were not qualified

Spiked Samples Analysis

Spiked and duplicate spiked sample analyses were performed on this data set All spike recoveries, with the exception of that for the lead spike on sample 98338710, were within the acceptance limits of +/- 25% Recovery of the noted spike was 63% Recovery from the duplicate spiked sample was acceptable and the average recovery, 74%, from this sample was marginal Data were not qualified based on this result on one out of three spiked samples, spiked samples were not analyzed for DTPA extractable metals or with the CEC analysis

Precision Data

The results of the spiked and duplicate spiked samples and duplicate sample results were used to evaluate precision on this sample set The relative percent difference (RPD) for all analytes was within the 20% acceptance window for duplicate analysis Mercury results for sample 98338710 had acceptable precision based on spiked sample results but not on duplicate sample results The duplicate may have been contaminated or the sample matrix non-homogenous Data for this sample was qualified J as estimated based on poor result precision

Serial Dilution

Serial dilutions were not analyzed with these samples

Laboratory Control Sample (LCS) Analysis

LCS analyses were within the windows established for each certified parameter, with the exception of results for elements other than arsenic and zinc, on one of the three LCS samples (M8245SL2) analyzed by ICP and GFAA The recoveries of cadmium, copper, nickel, and lead were - 134%, 126%, 127%, and 127% respectively, on the noted LCS sample Recovery of phosphorous was also higher from this sample, and phosphorous precision from this sample was poor Phosphorous level was not certified for the LCS sample Recoveries on these elements for this sample, were within 20% of the manufacturer's made to value level Data were not qualified based on the result on one out of three of the LCS samples for ICP and GFAA analysis

Appendix 2-H. Metals analysis quality assurance summary.

Data quality for this project met all quality assurance and quality control criteria with the exceptions that (1) recoveries of ICP and furnace elements were high for one of the three LCS samples, (2) recovery of lead from one of the spiked samples was low, and (3) replication of DTPA extractable copper and zinc from one sample was outside limits.

Because of several miscommunications at Manchester Laboratory, the mercury soil samples were not analyzed within the recommended holding time (28 days) for mercury in solid matrices. The lab does not feel that any mercury was lost from these samples before analysis, as the great majority of the samples are very dry and do not appear to have much biological or chemical activity associated with them. Additionally, the storage that these samples received (4°C, sealed, dark), indicate that loss of mercury would probably be minimal. However, we are qualifying the sample with J, denoting estimated values, recognizing that there is a possibility that some mercury may have been lost in storage.

To verify the stability of the samples and their mercury content, the lab will re-analyze the samples toward the end of October. This re-analysis will be free of charge. If the concentration has not substantially changed at this re-analysis, the lab will recommend that the J qualifiers be removed from the data set.

No other significant quality assurance issues were noted with the data. No certified reference materials were available for either DTPA extractable metals or for the cation exchange capacity (CEC). Spiked samples were not analyzed with these two methods.

Sample Information

The samples from the Metals in Soils study were received by the Manchester Laboratory on 08/1 8/98 and 8/26/98 in good condition.

Holding Times

All analyses, except those for mercury, were performed within the specified method holding times for metals analysis, 180 days for all metals except mercury. Mercury was analyzed at a time in excess of the 28-day holding time due to laboratory error. Mercury data were qualified J, as estimated, or UJ, as undetected at estimated detection level.

Instrument Calibration

Instrument calibration was performed before each analytical run and checked by initial calibration verification standards and blanks. Continuing calibration standards and blanks were analyzed at a frequency of 10% during the run and again at the end of the analytical run. All initial and continuing calibration verification standards were within the relevant method control limits. PLA calibration gave a correlation coefficient (r) of 0.995 or greater, also meeting method calibration requirements.

Appendix 2-G General chemistry quality assurance

Sample Information

Samples from the Metals in Soils study were received by the Manchester Laboratory on 8/18 and 8/26/98 in good condition Analysis for percent solids was performed immediately after sample arrival The samples were not stored in the freezer until TOC analysis could be performed due to the shorter turnaround time for this project

Holding Times

Soil TOC analysis, as well as pH analysis, was not performed within laboratory accepted holding times The TOC method in the Conventional Sediment Variables of the Puget Sound Protocols of March 1986 recommends that the samples should be stored frozen and can be held for up to 6 months There is no known established regulatory holding time for TOC sediment for samples that are stored at 4°C Due to pH probe drifting problems, the pH samples also were analyzed outside the laboratory established holding times There is also no known established regulatory holding time for this parameter

Instrument Calibration

Where applicable, instrument calibration was performed before each analysis, and verified by initial and verification standards and blanks All initial and continuing calibration verification standards were within the relevant EPA control limits All balances are calibrated yearly with calibration verification occurring monthly

Procedural Blanks

All procedural blanks were within acceptable limits

Precision Data

The results of the duplicate and triplicate analysis of samples were used to evaluate the precision on this sample set Relative percent differences (RPD) were within their acceptance windows of +/- 20% The relative standard deviations (RSD) were within their acceptance windows of +/- 20%

Laboratory Control Sample (LCS) Analyses

LCS and SRM analyses were within their acceptance windows of +/- 20%

Other Quality Assurance Issues

The results for the three pH duplicates have been qualified as estimates These samples were analyzed from leftover supernatant that was left on the counter overnight

Appendix 2-F. Methods summary.

Target Analysis	Method Reference
Cation Exchange Capacity	EPA SW-846 Method 9081
Total Available Phosphorus	EPA SW-846 Method 3050/6010
Extractable Metals (As, Cd, Cu, Pb, Zn)	Plant Available As, Cd, Cu, Pb, & Zn using DTPA Extraction followed by ICP Analysis (Spielman And Shelton, 1989)
Soil Particle Size	Conventional Sediment Variables: Particle Size (Puget Sound Estuary Program 1986)
pH	EPA SW-845 Method 9045C
Total Organic Carbon (TOC)	EPA SW-846 Method 415.1
Total Metals (Cd, Cu, Pb, Ni, Zn)	EPA SW-846 Methods 6010/6020 (analysis) Method 3050 (digestion)
Total Metals (As)	EPA SW-846 Method 7060 (GFAA)
Total Metals (Hg)	EPA SW-846 Method 7471 (CFAA)

and shrubs. This unit makes up about 6% of the county. It is about 50% Taunton soils, 40% Scoon soils, and the remaining 10% is components of minor extent. This unit is used mainly as rangeland and for irrigated crops and wildlife habitat. The production of forage is limited by restricted available water capacity. The main limitations for irrigated crops are the hazards of soil blowing and water erosion, restricted available water capacity, and steepness of slope.

Shano Soil Unit

This unit is in the southern part of the county. The native vegetation is mainly grasses and shrubs. The unit makes up about 4% of the county. It is about 95% Shano soils. and the remaining 5% is components of minor extent. Shano soils are on hills. The unit is used mainly for nonirrigated and irrigated crops and wildlife habitat. The main limitations for nonirrigated crops are the low annual precipitation and the hazard of water erosion. The main limitations for irrigated crops are the hazard of water erosion and steepness of slope.

Appendix 2-E. USGS soil type descriptions.

(Ames & Prych, 1984)

Kennewick-Warden-Sagemoor Soil Unit

This unit is located in the southern part of the county. The native vegetation is mainly grasses and shrubs. This unit makes up about 11% of the county. It is about 40% Kennewick soils, 20% Warden soils, and 10% Sagemoor soils. The remaining 30% is components of minor extent, such as Novark soils. This unit is used mainly for irrigated crops, rangeland, and wildlife habitat. The main limitations for irrigated crops are the hazards of soil blowing and water erosion and steepness of slope. The production of forage is limited by the low annual precipitation.

Timmerman-Quincy Soil Unit

This unit is located in the southern part of the county. The native vegetation is mainly grasses and shrubs; however, some areas are barren of vegetation. This unit makes up about 4% of the county. It is about 70% Timmerman soils and 15% Quincy soils. The remaining 15% is components of minor extent, such as Royal soils. This unit is used mainly for irrigated crops and wildlife habitat. The main limitations for irrigated crops are the hazards of soil blowing and water erosion, restricted available water capacity, and steepness of slope.

Ephrata-Malaga Soil Unit

This unit is in the southern part of the county. Native vegetation is grasses and shrubs. It makes up about 5% of the county, with 60% Ephrata soils, 35% Malaga soils, and the remaining 5% is components of minor extent. This unit is mainly used for irrigated crops and wildlife habitat. The main limitations for irrigated crops are restricted available water capacity, the hazard of water erosion, and steepness of slope.

Quincy Soil Unit

This unit is in the southern part of the county. This unit supports little if any native vegetation. This unit makes up about 12% of the county; 90% are Quincy soils and the remaining 10% is components of minor extent. This unit is used mainly as rangeland and for irrigated crops and wildlife habitat. The production of forage is limited by restricted available water capacity. The main limitations for irrigated crops are the hazard of soil blowing, restricted available water capacity, and steepness of slope.

Taunton-Scoon Soil Unit

This unit is in the southern part of the county. The native vegetation is mainly grasses

Appendix 2-D. Soil particle size distribution by soil type.

Sample Number	Soil Type	Soil Particle Size Distribution			
		% Gravel	*% Sand*	*% Silt*	*% Clay*
710	Warden silt loam	0.0	42.1	54.1	3.8
709	Warden silt loam	0.0	48.4	44.8	6.8
701	Warden silt loam	0.0	25.0	64.4	10.6
702	Shano silt loam	0.0	36.0	63.2	0.8
703	Shano silt loam	0.0	34.2	60.9	4.9
705	Shano silt loam	0.0	38.1	58.8	3.1
745	Scoon silt loam	0.9	65.3	33.7	0.1
723	Scoon silt loam	0.3	45.3	51.8	2.6
729	Novark silt loam	0.7	74.0	23.6	1.7
728	Novark silt loam	2.8	67.9	26.2	3.1
730	Kennewick silt loam	5.5	47.8	44.8	1.9
733A	Kennewick silt loam	0.0	0.0	0.0	0.0
737	Kennewick silt loam	0.0	50.0	45.8	4.2
719	Kennewick silt loam	0.8	65.3	31.3	2.5
744	Sagemoor silt loam	0.0	36.4	60.0	3.6
739	Sagemoor silt loam	0.0	48.2	48.7	3.1
746	Ephrata fine sandy loam	4.7	37.6	54.6	3.1
707	Ephrata fine sandy loam	10.2	45.2	40.6	4.0
717	Ephrata fine sandy loam	14.9	46.1	35.1	3.8
747	Kennewick fine sandy loam	0.1	57.8	41.6	0.5
738	Kennewick fine sandy loam	0.0	42.8	50.8	6.3
741	Royal very fine sandy loam	0.5	39.5	59.2	0.9
740	Royal very fine sandy loam	0.0	35.3	61.4	3.2
743	Prosser very fine sandy loam	1.5	35.9	61.1	1.5
742	Prosser very fine sandy loam	0.4	32.2	64.3	3.1
712	Quincy fine sand	0.5	72.4	26.3	0.8
711	Quincy fine sand	0.0	85.7	12.9	1.3
726	Quincy fine sand	0.0	85.2	14.6	0.2
725	Quincy fine sand	0.0	82.8	15.6	1.6
732	Timmerman coarse sandy loam	0.0	71.6	26.7	1.7
731	Timmerman coarse sandy loam	0.4	66.4	29.4	3.8
715	Timmerman coarse sandy loam	2.2	53.4	40.4	3.9
713	Timmerman coarse sandy loam	2.2	68.7	25.3	3.9

Appendix 2-C. Distance between agricultural sites and background sites.

Sample Number	Soil type	Proximity to Background site	Background Site Number
709	Warden silt loam	Adjacent	710
701	Warden silt loam	9 65 km	
703	Shano silt loam	Adjacent	702
705	Shano silt loam	1 61 km	
723	Scoon silt loam	6.44 km	745
728	Novark silt loam	Adjacent	729
733A	Kennewick silt loam	Adjacent	730
737	Kennewick silt loam	3.22 km	
719	Kennewick silt loam	33 8 km	
739	Sagemoor silt loam	8.05 km	744
707	Ephrata fine sandy loam	3.22 km	746
717	Ephrata fine sandy loam	24 1 km	
738	Kennewick fine sandy loam	9 65 km	747
740	Royal very fine sandy loam	Adjacent	741
742	Prosser very fine sandy loam	Adjacent	743
711	Quincy fine sand	Adjacent	712
725	Quincy fine sand	Adjacent	726
731	Timmerman coarse sandy loam	Adjacent	732
715	Timmerman coarse sandy loam	37 0 km	
713	Timmerman coarse sandy loam	40.2 km	

Appendix 2-B. Landowners' reasons for rejecting study participation.

None of the landowners contacted had heard of this study before our initial contact. Each contacted landowner was given the same information about the study and given the opportunity to ask questions or consult with others before deciding. Only two of the eight landowners who rejected the opportunity to participate opted to consult with other people prior to rejecting the opportunity. The other six rejections occurred during the initial contact. Those contacts were short, lasting only several minutes, with few questions from the landowners. Listed below are the reasons given for not choosing to participate in this study.

- "I don't trust the Government."
- "I don't like Ecology and this is against Cenex and the Farmers."
- "I don't like the idea of it."
- "This hits me cold, I don't have enough information about it, I might say yes if I had heard about it before."
- "I don't trust the government and I don't like some of the things they are doing like the nitrogen in the groundwater issue and talking about removing the dams for the salmon."
- "I have leased the farm and I don't think the operator would want you to trample the corn (unharvested corn field)."

Neither of the two landowners who took several days to decide gave a reason for not participating.

Appendix 2-A. Site selection criteria for metals in soils study.

Selection criteria for agricultural field sampling included (Holmgren et al., 1993):

- No obvious aerial deposition from industrial or automotive sources. Sites selected should be at least:
  - ✓ 8 km downwind from any stack emitter such as coal-fired generators, smelters, and foundries,
  - ✓ 200 m from US or state highways such as I-90,
  - ✓ 100 m from rural roadways,
  - ✓ 100 m from current, abandoned, or known obliterated building sites, and
  - ✓ 50 m from field boundaries.
- No known use of orchard pesticides such as lead arsenate; sites should be at least 8 km downwind of active orchard pesticides to minimize drifting.
- No known applications of biosolids or sewage sludge.
- If an appropriate matched non-agriculture site cannot be located, an agriculture site will be excluded.

Appendix 2-J. Graphical comparisons of other metals in soils studies.

The following graphs are comparisons of range and mean values of metal concentrations in soils from several studies (Ecology, 1994b; Holmgren *et al.*, 1993; and Ames and Prych, 1995).

Legend:

This Study:

- **CB98AG**: agricultural sample results
- **CB98BK**: background sample results

Natural Natural Background Soil Metals Concentrations in Washington State (Ecology, 1994b):

- **TCP-YB**: Yakima Basin results (Yakima, Kittitas, Chelan, and Grant counties)
- **TCP-GE**: Group E results (Benton, Spokane, Lincoln, Adams, Okanogan, and Whitman counties)

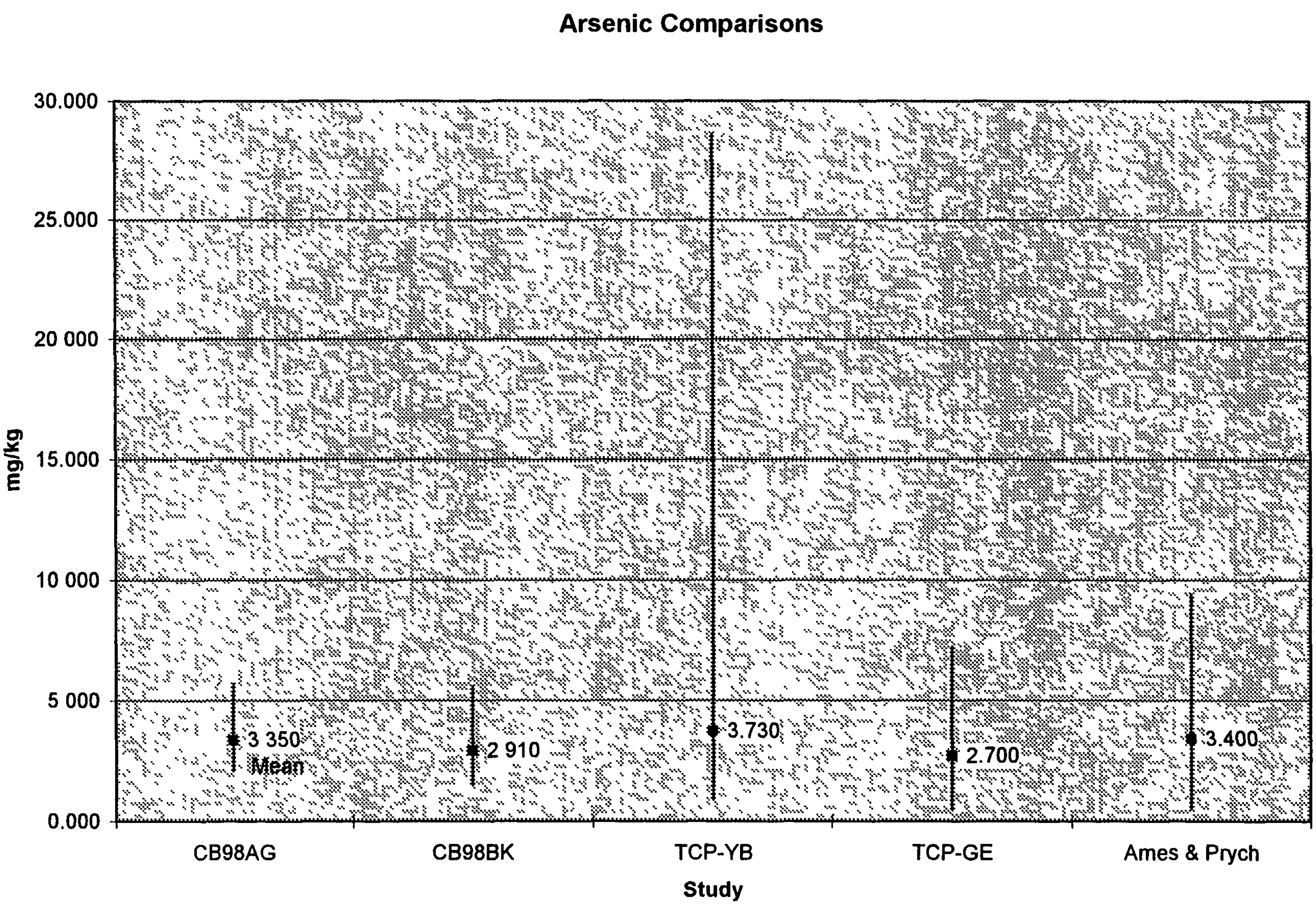
Background Concentrations of Metals in Soils from Selected Regions in the State of Washington (Ames & Prych, 1995):

- **Ames & Prych**: results from "total-recoverable" method

Cadmium, Lead, Zinc, Copper, and Nickel in Agricultural Soils of the United States of America (Holmgren *et al.*, 1993):

- **Holmgren**: results from Grant and Adams Counties only

Vertical lines represent the range of values for that study; boxes (■) represent arithmetic means.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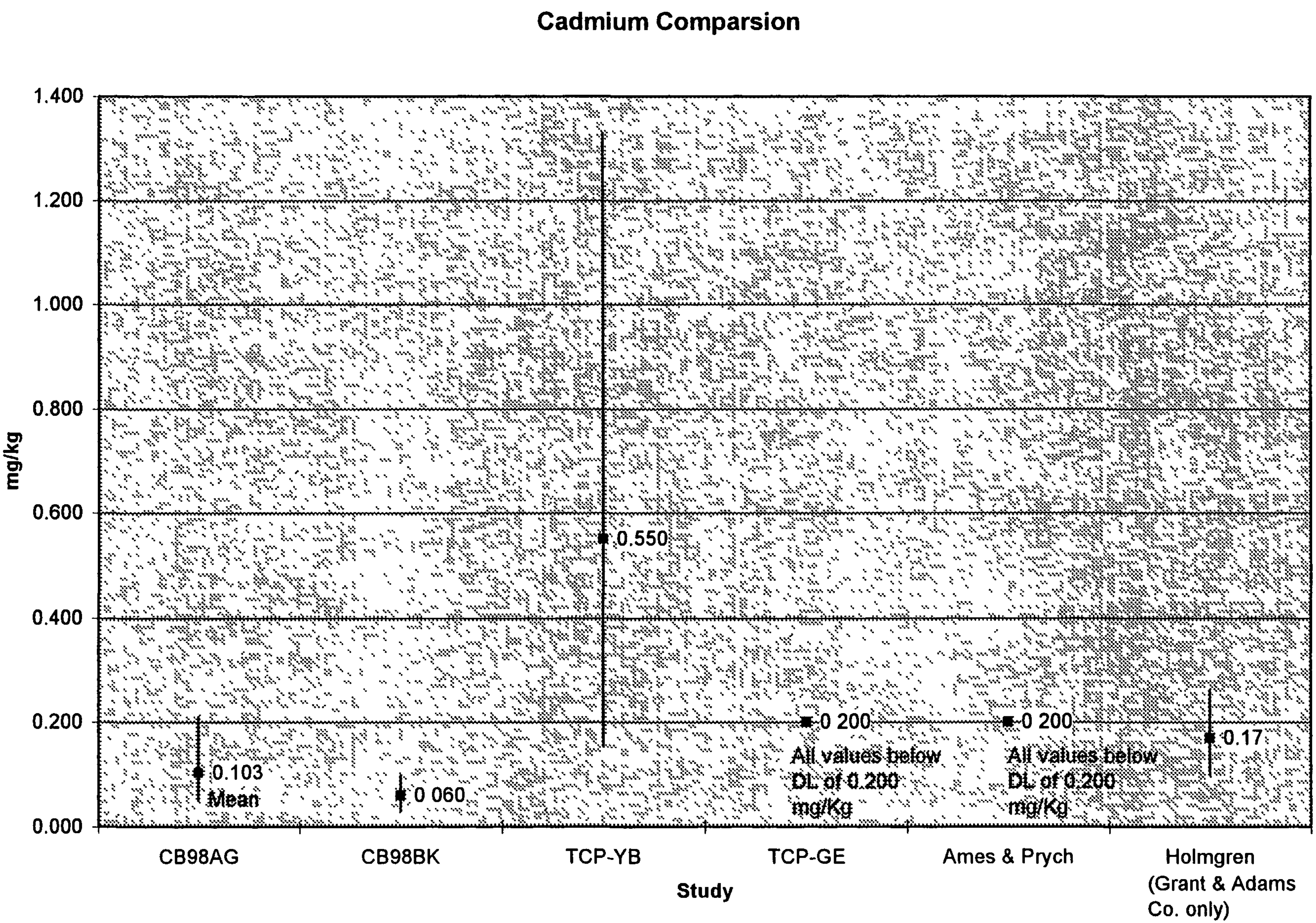
25.000
20 000
15.000
10 000
5 000
0.000
mg/kg
3 350
Mean
2 910
3.730
2.700
3.400
CB98AG
CB98BK
TCP-YB
TCP-GE
Ames & Prych
Study


Cadmium Comparsion
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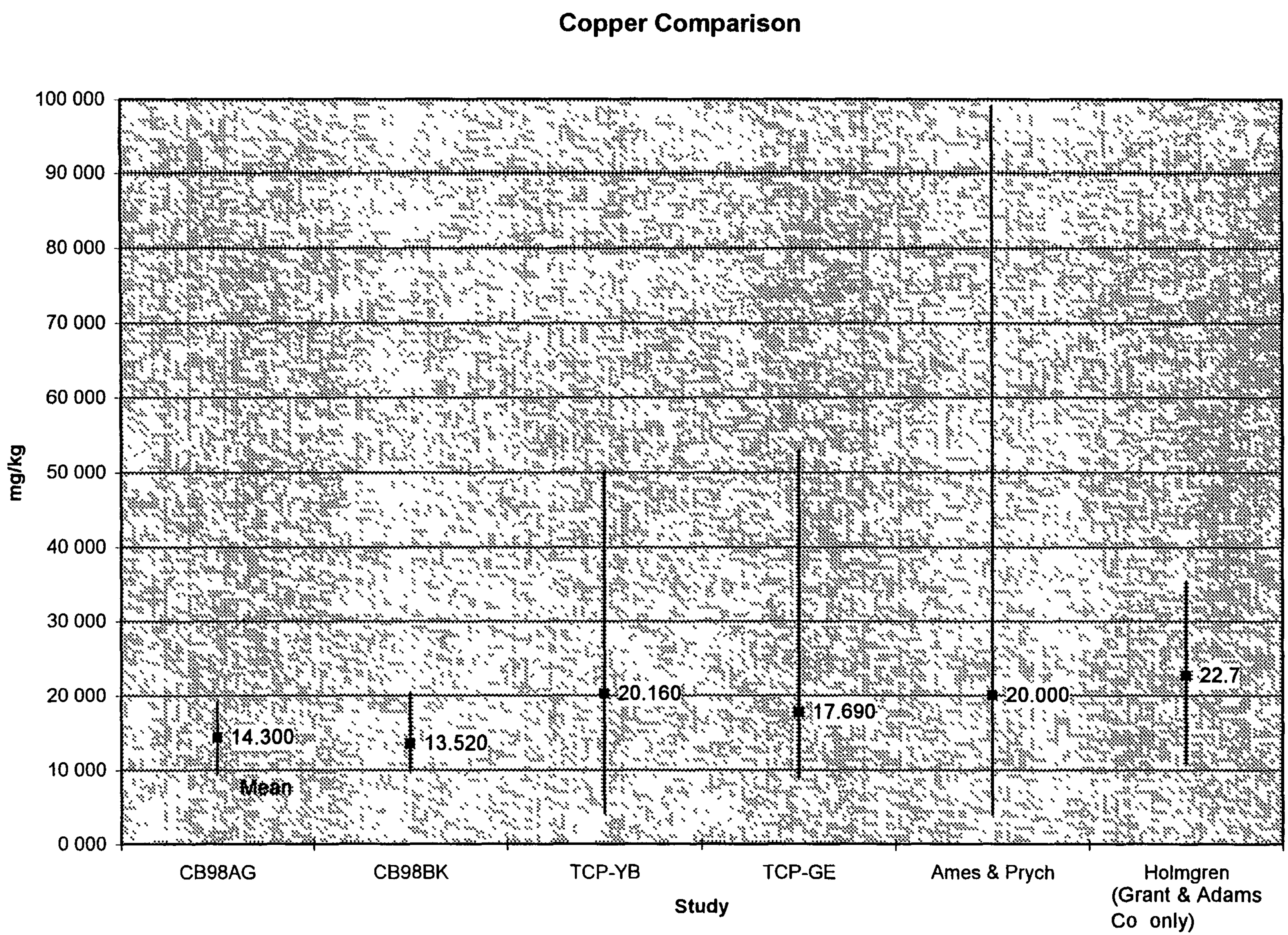
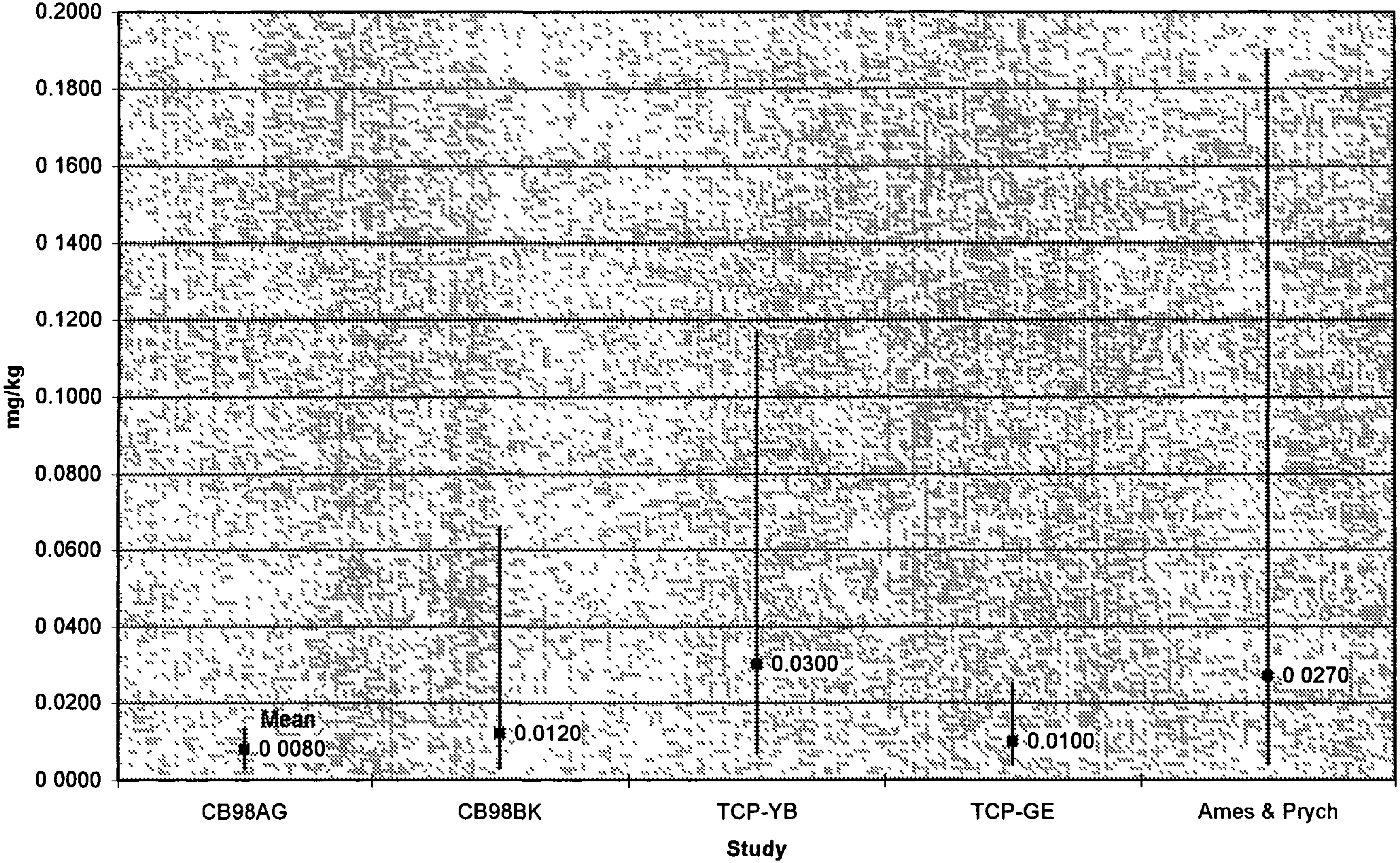
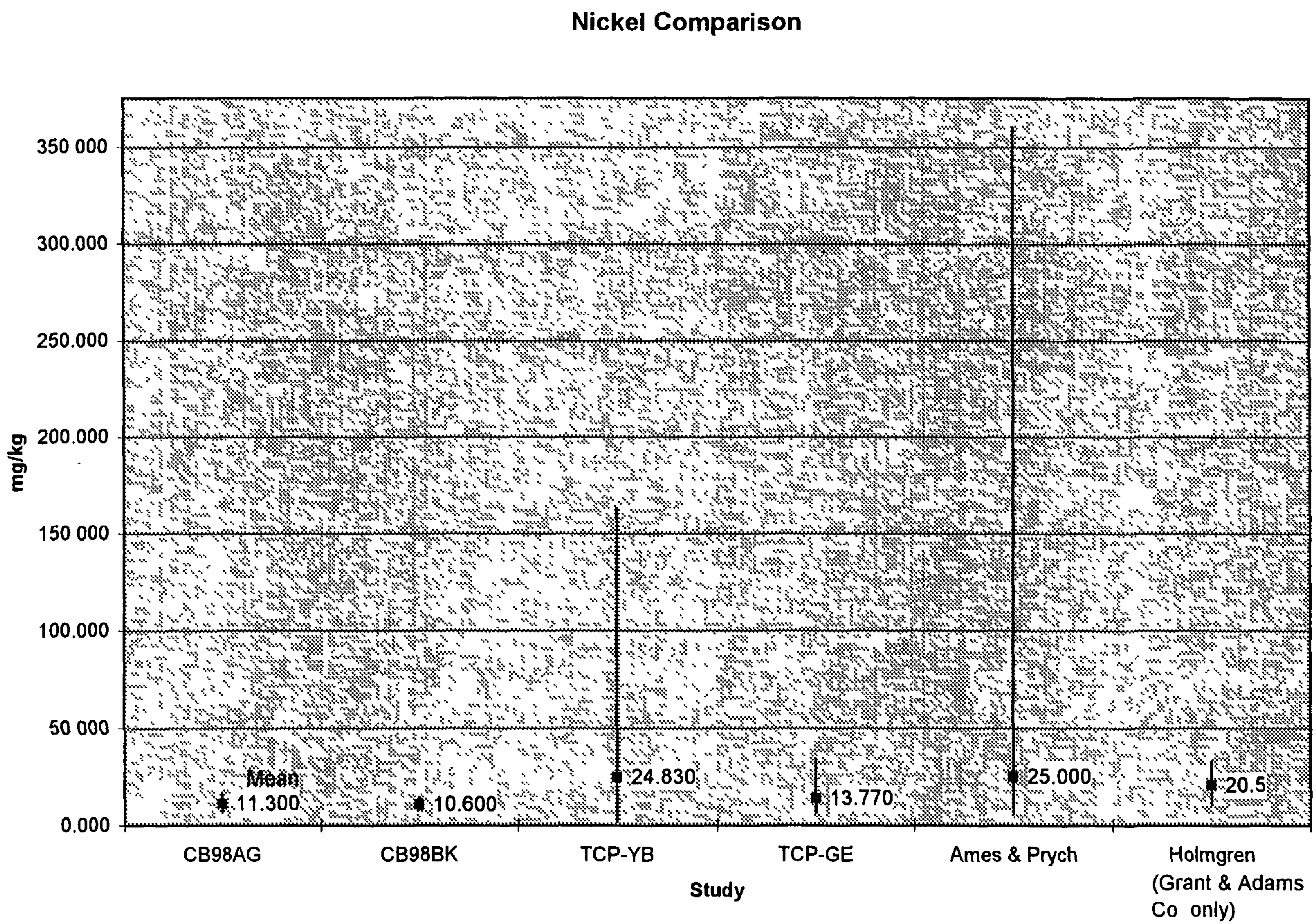
1.200
1.000
0.800
0.600
0.400
0.200
0.000
mg/kg
0.103
Mean
0 060
0.550
0 200
All values below DL of 0.200 mg/Kg
0 200
All values below DL of 0.200 mg/Kg
0.17
CB98AG
CB98BK
TCP-YB
TCP-GE
Ames & Prych
Holmgren (Grant & Adams Co. only)
Study

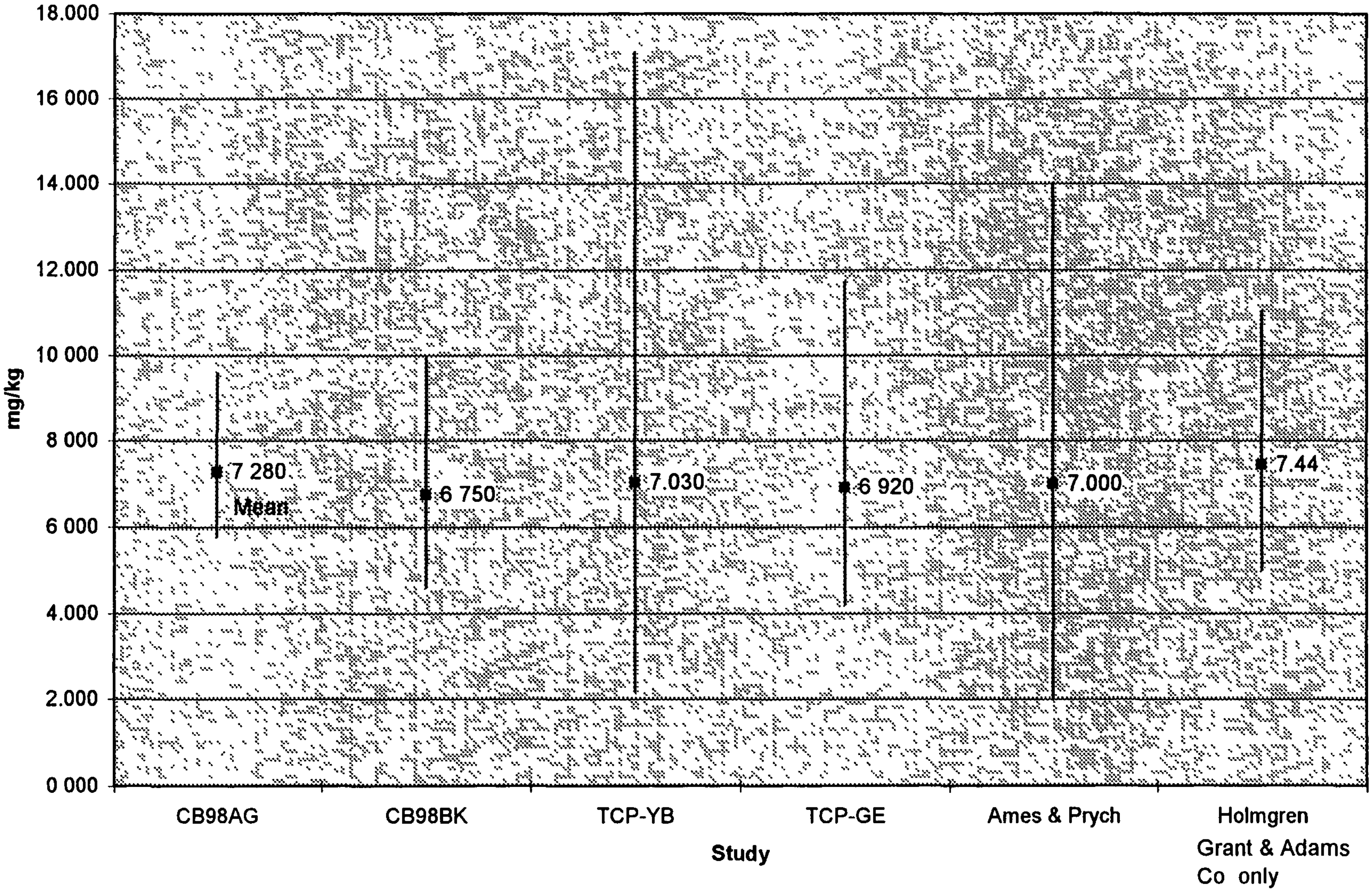

Copper Comparison
100 000
90 000
80 000
70 000
60 000
50 000
40 000
30 000
20 000
10 000
0 000
mg/kg
14.300
Mean
13.520
20.160
17.690
20.000
22.7
CB98AG
CB98BK
TCP-YB
TCP-GE
Ames & Prych
Holmgren (Grant & Adams Co only)
Study

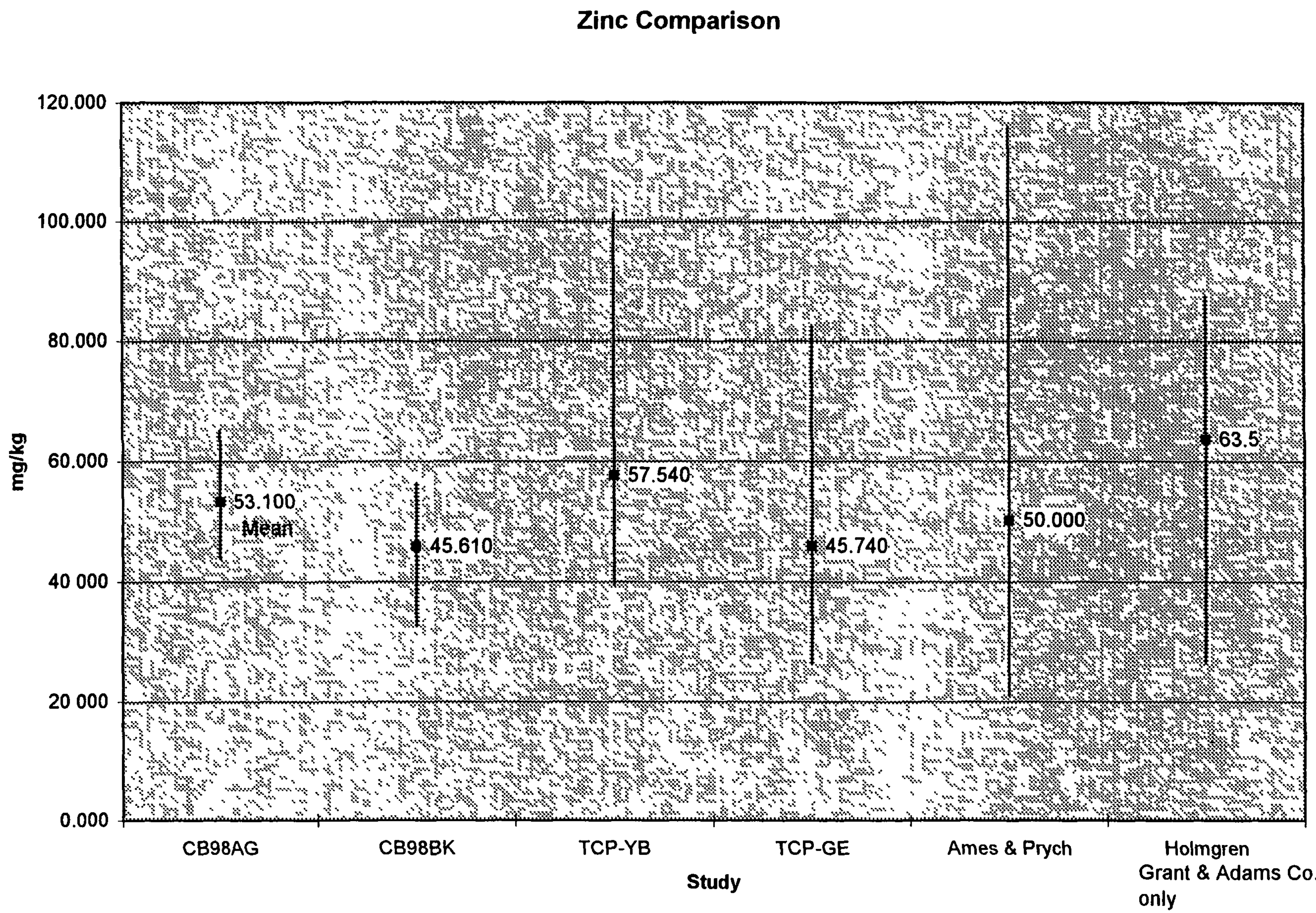
Mercury Comparison




Nickel Comparison
mg/kg
350.000
300.000
250.000
200.000
150.000
100.000
50.000
0.000
Mean
11.300
10.600
24.830
13.770
25.000
20.5
CB98AG
CB98BK
TCP-YB
TCP-GE
Ames & Prych
Holmgren (Grant & Adams Co only)
Study

Lead Comparison




Zinc Comparison
120.000
100.000
80.000
60.000
40 000
20 000
0.000
mg/kg
53.100
Mean
45.610
57.540
45.740
50.000
63.5
CB98AG
CB98BK
TCP-YB
TCP-GE
Ames & Prych
Holmgren
Grant & Adams Co.
only
Study

Appendices

for

3. Dioxins in Soils

Appendix 3-A. Urban area dioxin soil sampling random allocation.

Urban Name	Land area (sq km)	Population	Number of Samples Allocated
Bellingham	77.69	59,317	0
Bremerton	143.04	112,977	0
Longview	86.57	54,985	0
Olympia	143.42	95,471	0
Vancouver	174.23	167,482	0
Richland-Kennewick-Pasco	251.78	116,118	2
Greater Seattle (incl. Everett)	1,522.63	1,744,086	9
Spokane	294.19	279,038	1
Tacoma	603.04	497,210	2
Yakima	96.92	88,054	0
Total	3,393.51	3,214,738	14

Appendix 3-B. Quality Assurance Memos for dioxin in soils data.

Appendix 3-B is included in a supplemental report, Ecology Publication 98-332:

<u>Supplementary Appendices: Preliminary Screening Survey of Metals and Dioxins in Fertilizers, Soil Amendments, and Soils in Washington State</u>

Appendix 3-C. Dioxin in soils results and TEQ calculations.

Appendix 3-C is included in a supplemental report, Ecology Publication 98-332:

Supplementary Appendices: Preliminary Screening Survey of Metals and Dioxins in Fertilizers, Soil Amendments, and Soils in Washington State

Appendix 3-D. TEQ values of soil samples collected from selected Washington State land use areas (ng/kg).

Land Use	TEQ ND = 0	TEQ ND = 1/2 DL	TEQ ND = DL	Lab #
Forested Lands				
East non-commercial	5.16	5.57	6.04	328341
East non-commercial	0.449	1.60	2.76	338331
West non-commercial	4.93	5.69	6.46	308000
West non-commercial	2.57	4.86	7.15	318241
East commercial	0.0330	1.05	2.06	338330
East commercial	0.914	3.84	6.76	318243
West commercial	2.02	2.70	3.38	328332
West commercial	2.42	2.80	3.17	338333
Open Areas				
East rangeland grazed	0.0431	0.891	1.74	338332
East rangeland grazed	0.0400	1.31	2.59	328336
West rangeland grazed	0.617	1.40	2.19	308004
West rangeland grazed	4.59	5.87	7.15	328331
East non-grazed	0.0460	0.631	1.22	328335
East non-grazed	0.0834	1.36	2.64	328340
West non-grazed	2.37	2.87	3.37	328330
West non-grazed	0.330	1.09	1.84	318242
Urban Areas				
Richland	4.75	7.09	9.44	328337
Kennewick	1.08	1.92	2.76	328339
Spokane	0.984	3.00	5.01	328333
Tacoma 1	19.5	21.9	24.4	318239
Tacoma 2	9.47	11.7	13.9	318240
Seattle 1	0.313	0.699	1.08	318230
Seattle 2	5.13	5.47	5.81	318238
Seattle 3	4.72	5.78	6.84	318236
Seattle 4	0.133	0.639	1.14	318231
Seattle 5	0.804	1.21	1.62	318235
Seattle 6	2.10	3.02	3.94	318232
Seattle 7	0.729	1.52	2.30	318233
Seattle 8	5.96	6.31	6.66	318234
Seattle 9	1.36	2.81	4.26	318237
Duplicate Samples				
Spokane	0.326	4.36	8.39	328334
Richland	4.50	8.26	12.0	328338

ND = Non-detect
DL = Detection Limit
ND = 0: if congener not detected, concentration assumed = 0
ND = ½ DL. if congener not detected, concentration assumed = ½ detection limit
ND = DL. if congener not detected, concentration assumed = detection limit

Appendix 3-E. Apparent percent grain size and percent total organic carbon (TOC) of soil samples from selected Washington State land use areas.

Land Use	Gravel	Sand	Silt	Clay	TOC 70	TOC 40	Lab #
Forested Lands							
East non-commercial	11.2	59.5	26.9	2.4	8.06	9.48	328341
East non-commercial	13.8	72.7	12.0	1.5	22.9	25	338331
West non-commercial	0.0	91.4	8.4	0.2	43.6	60.6	308000
West non-commercial	10.3	66.8	17.6	5.2	41.1	45.9	318241
East commercial	2.7	56.1	39.3	1.8	6.23	6.81	338330
East commercial	2.8	53.5	42.1	1.6	6.58	7.69	318243
West commercial	40.6	51.1	7.8	0.5	8.16	9.45	328332
West commercial	37.7	60.0	1.4	0.9	11.7	13.5	338333
Open Areas							
East rangeland grazed	10.8	50.1	36.7	2.3	1.32	1.42	338332
East rangeland grazed	3.3	32.6	60.9	3.2	1.95	2.27	328336
West rangeland grazed	1.3	53.7	43.4	1.6	6.54	7.83	308004
West rangeland grazed	16.7	74.6	8.4	0.3	7.92	8.98	328331
East non-grazed	0.0	53.9	43.9	2.2	3.91	4.52	328335
East non-grazed	15.4	67.1	15.3	2.2	9.05	11.5	328340
West non-grazed	25.6	63.6	10.4	0.3	11.1	12.5	328330
West non-grazed	1.8	77.2	16.3	4.7	39.5	44.5	318242
Urban Areas							
Richland	0.1	67.3	30.5	2.2	3.97	4.69	328337
Kennewick	2.6	69.3	26.4	1.7	2.51	2.76	328339
Spokane	0.8	54.2	43.0	2.0	5.56	6.21	328333
Tacoma	5.1	78.4	16.1	0.4	5.50	6.13	318239
Tacoma	5.6	73.5	19.7	1.2	7.08	7.86	318240
Seattle 1	4.2	89.9	5.4	0.5	0.95	1.03	318230
Seattle 2	4.1	77.4	18.1	0.4	5.77	6.45	318238
Seattle 3	0.7	77.6	21.5	0.2	3.82	4.25	318236
Seattle 4	10.8	87.7	1.1	0.5	0.17	0.18	318231
Seattle 5	5.6	89.0	5.3	0.1	4.17	4.35	318235
Seattle 6	10.8	78.8	10.1	0.2	5.61	6.23	318232
Seattle 7	13.4	76.6	9.6	0.4	2.47	2.75	318233
Seattle 8	3.8	85.9	10.0	0.4	4.64	5.17	318234
Seattle 9	7.9	79.9	10.4	1.7	3.28	3.63	318237
Duplicate Samples							
Spokane	3.4	55.9	39.2	1.6	5.95	6.91	328334
Richland	4.9	64.8	28.2	2.2	4.07	4.37	328338